SPECIAL REPORT 97-26

Antifreeze Admixtures for Concrete

Charles J. Korhonen, Edel R. Cortez, Timothy A. Durning, and Ara A. Jeknavorian

October 1997

Abstract: The goal of this project was to develop a chemical admixture that would reduce the need for wintertime thermal protection of freshly placed concrete. Chemicals were investigated for their ability to promote strength gain in concrete cured below 0°C. The project was carried out in five phases. Phase 1 evaluated existing and new admixtures. Phase 2 measured the effect of promising chemicals on concrete properties. Phases 3 and 4 tested the practicality of using the new technology/admixture in the field. Phase 5 disseminated the findings through an Army conference and through the development of this report, in addition to normal W.R. Grace advertising channels. Laboratory strength tests established that two prototype admixtures were capable of protecting concrete down to –5°C. Results from other laboratory tests show that the chemicals pose no harm to the concrete or embedded ferrous metals. Concrete containing the prototype admixtures passes standard freeze–thaw tests, does not shrink unusually, does not contain harmful alkalis, and does not produce irregular hydration products. Field tests clearly demonstrated that working with these new admixtures requires no new skills. The concrete can be mixed at lower temperatures, saving energy. The admixtures are easily dosed into the mixing trucks, as is normal practice today, and concrete is finished in the usual manner. Estimates show that the two prototype admixtures can extend the construction season by as much as three months in the contiguous United States. The prototype has proved that low-temperature admixtures are possible. The industry partner sees the need to develop admixtures that will work to –10°C before going commercial with this technology.

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US Army Corps
of Engineers®
Cold Regions Research &
Engineering Laboratory

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and Ara A. Jeknavorian

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Prepared for
OFFICE OF THE CHIEF OF ENGINEERS

PREFACE

This report was prepared by Charles J. Korhonen and Edel R. Cortez, Research Civil Engineers, Civil Engineering Research Division, Research and Engineering Directorate, U.S. Army Cold Regions Research and Engineering Laboratory (CRREL), and by Timothy A. Durning, Project Manager, and Ara A. Jeknavorian, Chemical Scientist, W.R. Grace & Co. (WRG).

This research project was conducted under the authority of the U.S. Army Corps of Engineers Construction Productivity Advancement Research (CPAR) program. The project was titled *Antifreeze Admixtures for Concrete* and was approved in August 1991. It was conducted in partnership between CRREL and WRG. The research work was conducted from November 1991 to December 1994.

Technical review of this report was provided by Ken Rear, Technical Services Manager, and Charles I. Sanders, Jr., Manager of Analytical and Technical Services Laboratory, WRG. Although many individuals from both the Corps of Engineers and WRG supported this research work in various ways, the authors acknowledge the support of Kevin Grogan, Neal Berke, Mauro Scali, and Ken Nelson of WRG; Brian Charest, Charles Smith, and Patrick Black of CRREL; and Stanley Jacek and Kurt Romisch of the Corps of Engineers, Sault Ste. Marie Area Office.

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Antifreeze Admixtures for Concrete

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INTRODUCTION

Background

The American Concrete Institute (ACI) is the technical authority on concrete technology in the United States. In its guidance for cold weather concrete (American Concrete Institute 1988), ACI outlines procedures to prevent early-age freezing, to ensure adequate strength for safe removal of form work, and to avoid thermally induced cracking. In order to produce quality concrete, several parameters must be carefully controlled. The air temperature as well as the concrete temperature needs to be monitored constantly before and after casting to avoid cold and hot spots. Despite careful control, it is not uncommon to find excessively cold and hot areas in the same enclosure at the same time. Neither is it uncommon to find spots where hot air has dried out the fresh concrete. Local dehydration and significant temperature gradients can result in concrete of nonuniform properties and in concrete that is thermally cracked.

If combustion heaters are used, carbonation of the concrete surface may occur and cause soft surfaces and surface crazing. Carbon monoxide from partial combustion presents a hazard to workers. The risk of uncontrolled fire exists wherever open flame heaters are used. Provisions for these procedures date back to the 1930s. Basically, the conventional practice today is to artificially warm the environment where concrete is mixed, cast, and cured, keeping it at or above 5°C. The high cost of thermal protection discourages winter construction. Underutilization of resources and seasonal unemployment are common among concrete practitioners in cold regions.

One alternative to thermal protection methods is the use of admixtures that allow fresh concrete to achieve acceptable strength when cured in cold environments. These admixtures are variously known as antifreeze admixtures, low-temperature admixtures, or freeze-protection admixtures. The term "antifreeze admixtures" is adopted in this report to convey the implication that they work at temperatures below the freezing point of water. The concept of antifreeze admixtures for concrete is found in foreign literature reporting early experiences in Scandinavian countries and the former Soviet Union (Korhonen 1990). With antifreeze admixtures, there is little need for building enclosures, insulation, or heaters. The properties of the concrete are more uniform, and thermal gradients are insignificant. Concerns about accidental early-age freezeups are diminished because the internal temperature of the concrete can be below 0°C. Antifreeze admixtures have two purposes: to depress the freezing point of water and to accelerate the hydration of cement at low temperature.

This research project combined the expertise of two organizations: the U.S. Army Cold Regions Research and Engineering Laboratory (CRREL), which has studied low-temperature admixtures since 1990 (Borland 1994a, b; Korhonen et al. 1994a, b; Korhonen et al. 1995) and W.R. Grace (WRG), which is a leading producer of admixtures for concrete, with extensive experience with concrete accelerators.

Project objectives

The objectives of this project were to

- Evaluate the low-temperature strength performance of Daraset, a commercial concrete strength accelerator.
- Develop new cold weather admixtures.
- Recommend changes in construction standards.

The performance of Daraset at warm temperatures was well known, but its performance below 0°C was not. Daraset was tested along with other

Table 1. Research work organization.

Phase no.	*Description*
1	Effect of chemicals on strength gain
2	Evaluation of best admixtures
3	Prototype slab-wall application
4	Field application of new admixtures
5	Technology transfer

chemical formulations in search of an admixture that would cause concrete to develop acceptable strength at temperatures below 0°C. Because current concrete construction standards cover concrete work down to 5°C, the overall goal was to produce an admixture that would promote concrete strength at –5°C to meet or exceed the strength of admixture-free concrete at 5°C.

Another objective of this project was to recommend changes that integrate the findings of this research into relevant construction standards. It was the intention of the industry partner to develop an admixture that would provide adequate strength at a sufficiently low temperature to justify the investment involved in the implementation of a new product line.

Approach

The research work was organized into five phases (listed in Table 1). Phase 1 evaluated a large number of potential chemical compounds for their ability to promote concrete strength at –5°C. The admixtures that provided the best strength performance at low temperature were selected for Phase 2. This phase was a more comprehensive evaluation that included testing to determine the effect of these chemicals on relevant concrete properties. Phase 3 consisted of a prototype concrete application geared to explore practical job site issues such as placement, finishing, and curing of full-size concrete elements. Phase 4 integrated the experience gained in the prototype application into an actual field application built under winter conditions; this demonstrated the advantage of using antifreeze admixtures over conventional thermal protection methods. Phase 5 consists of technology transfer efforts, such as this report.

Table 2. Concrete mix design.

Cement type	ASTM C150, Type I
Cement content	362.8 kg/m^3 for tasks 1A to 1F 418.6 kg/m^3 for task 1H
Water/cement ratio	0.48, or as indicated
Target slump	5 cm
Aggregate gradation	ASTM C 33, # 6
Aggregate source	Lebanon, New Hampshire

LABORATORY EXPERIMENTS

Phase 1: Effect of chemicals on strength gain

Objective

To develop a chemical formulation that would allow concrete cured at –5°C to gain strength at least as fast as control concrete cured at 5°C.

Experimental approach

Early experiments with single chemical compounds, experiences found in the literature, physico-chemical data available for each chemical compound, and knowledge of the chemistry of cement hydration formed the basis for the formulation of candidate admixtures. The candidate admixtures were made of mixtures of two, three, or four chemical compounds. These formulations were tested for their ability to perform as antifreeze admixtures, i.e., chemicals that depress the freezing point of water and accelerate the hydration of cement.

The experimental work began by screening a set of chemical formulations using a strength gain criterion. A series of concrete mixes were made (Table 2), each including one candidate admixture at one of various dosages. A number of cylinders were cast and cured under one of several temperatures using special coldrooms. Additionally, control concrete (admixture free) was mixed, cast, and cured under the same conditions as the admixtured specimens (Table 3). At the prescribed time, the cylinders were transported out of their coldrooms and allowed to thaw up to a controlled temperature of about 10°C at their center of mass. This was a necessary precaution to avoid testing

Table 3. Test parameters.

Chemical admixtures	As needed	
Curing temperatures	4	(20°C, -5°C, -10°C and -20°C)
Testing ages	3	(7, 14, and 28 days)
Mix size	0.04 m^3	
Specimen replicates	3	
Specimen size	7.6 cm × 15.2 cm	Cylinders

Table 4. Experimental tasks in Phase 1, and number of mixes.

Task	*Mixes*
1A	20
1B	20
1C	20
1D	8
1E	5
1F	10
1Ga	10
1Gb	5
1Gc	8
1H	7

frozen concrete, which could yield false high strengths that vanish upon thawing. The cylinders were compression tested at various ages.

Phase 1 was divided into ten tasks (Table 4). Each task consisted of testing one set of chemical formulations. The objective was to identify chemical formulations that best promote the strength of concrete cured at low temperatures. Slump, slump loss, and set times were also measured for several mixes. Retarders and some adjustments to the mix design were implemented in later tasks to obtain an adequate compromise between strength and workability.

With the exception of Task 1G*, the specimens were concrete cylinders (7.6 × 15.2 cm) cast in plastic molds. The cylinders were kept sealed in their plastic molds until ready for compression testing, and then were cast at room temperature (hereby defined as approximately 20°C) and brought to their corresponding curing room within 40 minutes after water was added to the mix.

On the date of their compression test, the corresponding cylinders were brought to room temperature environment, demolded, and allowed to stand just long enough to ensure that no specimen would contain ice during testing. Typically, a cylinder would be allowed to reach 10°C before testing. Replicate dummy cylinders containing a thermocouple at their center of mass were also cast to monitor temperatures.

Mixing time and sequence

Mixing procedures followed ASTM C 192, "Standard Method of Making and Curing Concrete Test Specimens in the Laboratory," specifically paragraph 6.1.2:

1. Add coarse aggregate.
2. 1/3 mix water (admixture free), start mixer.
3. Fine aggregate (stop mixer if needed).
4. Cement (stop mixer if needed).
5. Remaining water containing admixture.
6. Mix for three minutes, stop mixing for three minutes, mix for two minutes.
7. Discharge.

* Task 1G was conducted on mortar.

Phase 1, Tasks 1A to 1H

Phase 1 contained ten tasks. During the early planning stages of this project, it was envisioned that the search for a –5°C admixture would require two or at most three tasks. However, we soon learned that a compromise between low-temperature strength development and workability of the fresh concrete was needed in order for the admixture to be practical. New admixtures were formulated to address these issues and testing continued. Next, differences in low-temperature (–5°C and –10°C) performance indicated a need to reformulate some of the early admixtures to reach a balance between strength acceleration and freeze-point depression. Finally, after ten tasks, two admixtures code-named DPTC* and DP were selected for further study in Phase 2.

In each task, a set of concrete mixes, each containing a given chemical admixture at a given dosage, was mixed, and cylinders were cast, cured, and compression-tested to determine the effect of each admixture on the strength development of concrete cured at various temperatures.

All Phase 1 tasks were conducted on concrete cylinders, except tasks 1Ga, 1Gb, and 1Gc, which were conducted on mortar specimens. Mortar was used to expedite the study of a series of admixtures.

Experimental data

The data from only certain tasks will be discussed in this report. For those wishing more detail, the test data for all ten Phase 1 tasks are presented in Appendix A. There, "N/A" stands for "not available," and it appears wherever a mix number was not used in that particular task, or the test was limited according to judgment of relevance. For tasks with fewer than 20 mixes, the charts show a blank space where a mix number was not used. The blank spaces in the charts were included to keep a consistent format that facilitates comparison. The admixture code name, dosage, and water/cement ratio for each mix are given in separate tables. Code names were used rather than chemical formulations where there was a need to preserve the industry partner's proprietary rights.

The ACI 306 specifications for cold weather concrete are valid for concrete cured at 5°C or above. In this project, an antifreeze admixture was required to deliver a seven-day strength for speci-

* The chemical compositions are disclosed in Table 5 for the selected admixtures, except for those protected by the industry partner's proprietary rights.

Table 5. Chemical composition of best admixtures tested in Phase 1.

Mix no.	*Antifreeze admixture formulation*	*Dosage (% CWT)*	*Water/cement ratio*
1A_1*	Control admixture-free	0	0.48
1A_20	KC1 (3 parts of sodium nitrate + 1 part of sodium sulfate)	8.0	0.48
1B_19	CCSN	4.0	0.48
1D_4	K_2CO_2 + lignosulfonate	6.0/1.5	0.38
1D_7	$Ca(NO_2)_2$ + $NaNO_2$	3.0/3.0	0.48
1E_5	CM-48†	6.0	0.48
1E_3	CM-42†	6.0	0.48
1F_8	$Ca(NO_2)_2$ + WRDA-19**	4.0/0.7	0.40
1H_2	DP†	6.0	0.43
1H_6	DPTC†	6.0	0.43

* 1A_1 means mix number 1 in task 1A.
† Code name used only to protect proprietary rights.
** WRDA-19 is a high-range water-reducing admixture commercially available from W.R. Grace, Inc.

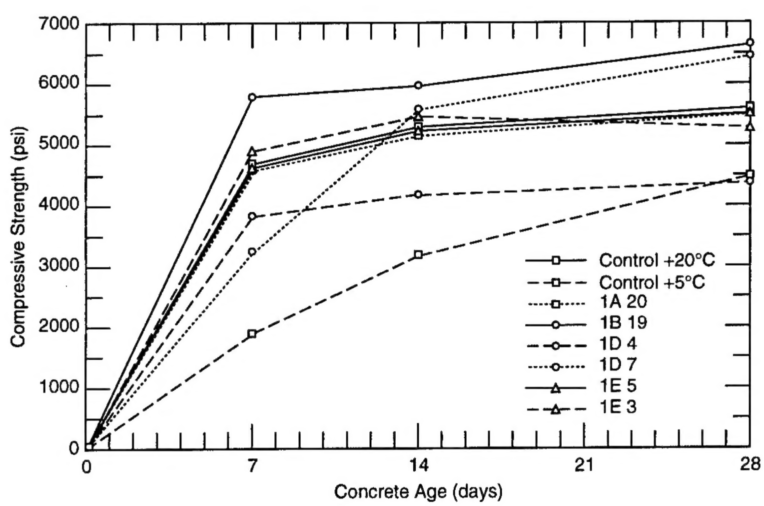
mens cured at –5°C that was equal to or better than that of nonadmixture concrete cured at 5°C. The admixtures that developed higher strength at –5°C were selected for further testing in Phase 2. It is important to note that these temperatures are at the center of mass of concrete cylinders. In actual concrete structures, the air temperature can be significantly lower without harming the concrete because of the effect of the internal heat of hydration.

Analysis of Phase 1 test results

The strength performances of the selected admixtures are presented in the figures below. The selection criteria at this stage were chiefly based on the strength developed at seven days of cure at –5°C. In these graphs each mixture is identified by a code. The first two characters indicate the task number for which the test results were obtained followed by the mix number used in the corresponding task. Figures 1a through 1d display the test results at 20°C, –5°C, –10°C, and –20°C. Two control curves corresponding to admixture-free concrete are included for reference. One corresponds to concrete cured at 20°C. The second corresponds to concrete cured at 5°C, the lowest temperature currently covered by the ACI 306 specification. Table 5 shows the chemical composition of the selected admixtures. The admixtures subject to proprietary rights are identified by their code name only.

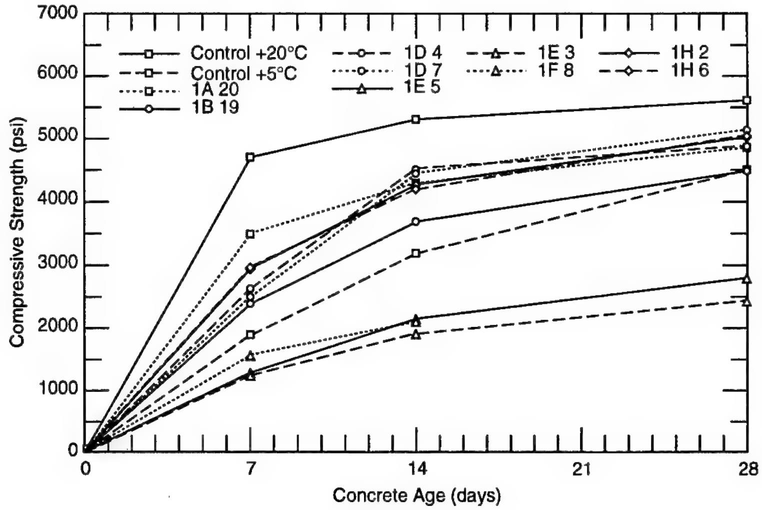
Figure 1a, corresponding to a curing temperature of 20°C, shows that CCSN (Mix No. 1B_19) enhanced the strength of concrete for at least the first 28 days. The admixture $Ca(NO_2)_2$ + $NaNO_2$ (mix 1D_7) caused lower strength at seven days, but higher strength at 14 days and thereafter. The admixture K_2CO_2 + lignosulfonate (mix 1D_4) reduced the strength at all times, at least up to 28 days. The rest of the admixtures did not have a significant effect at room temperature.

Figure 1b, corresponding to a curing temperature of –5°C, shows that most of the selected admixtures allowed concrete strengths to be between the values for the 20°C and 5°C control concrete. Any strength developed at low temperature that

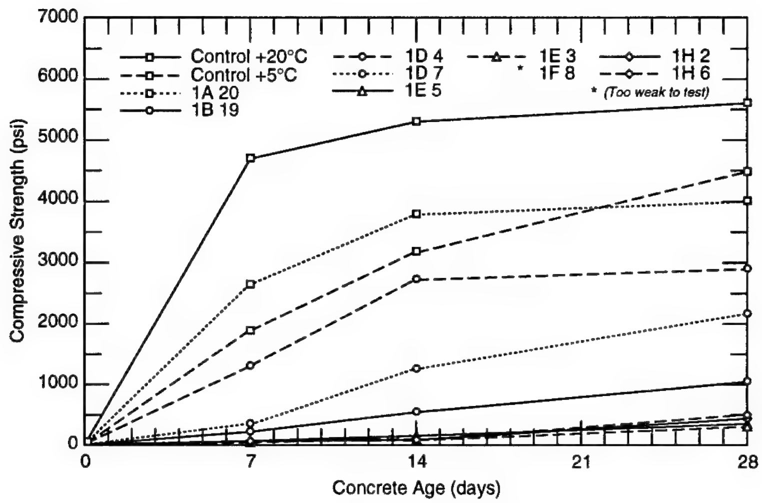


a. 20°C.

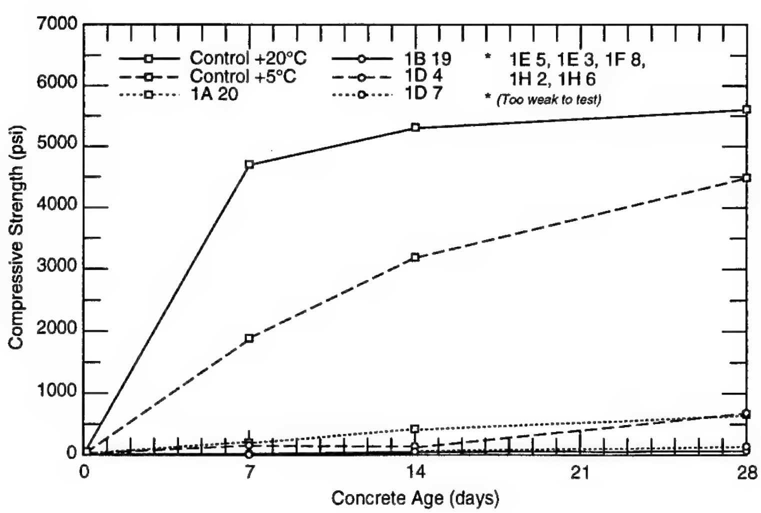
Figure 1. Compressive strength of best admixtures of Phase 1 cured at various temperatures. Mixes 1F_8, 1H_2, and 1H_6 were not tested at 20°C.



b. –5°C.



c. –10°C.



d. –20°C.

Figure 1 (cont'd).

equals or exceeds that of the control concrete cured at 5°C for seven days and for 14 days is regarded as acceptable. Three admixtures fell below these acceptance limits: CM-42 (mix 1E_3), $Ca(NO_2)_2$+ WRDA-19 (mix 1F_8), and CM-48 (mix 1E_5).

Figure 1c, corresponding to a curing temperature of –10°C, shows that only the KC1 admixture (mix 1A_20) developed strengths higher than the acceptance limit. However, its 28-day strength fell below that of the control concrete cured at 5°C. The admixture K_2CO_2 + lignosulfonate (mix 1D_4) came close to the acceptance band. The strengths of concrete made with all other admixtures were lower than the acceptance limit. Figure 1d, corresponding to a curing temperature of –20°C, shows that none of the admixtures provided acceptable strength at this temperature.

The admixtures tested in the ten tasks of Phase 1 were formulated based on experiences found in the literature, preliminary tests conducted by the partner organizations prior to this project, physical and chemical data available for the chemicals involved, and the researchers' knowledge of cement chemistry. The admixture KC1 was developed at CRREL before this CPAR project began. KC1 was included in this project as a benchmark for other admixtures, and to expand the experimental data on its performance. A U.S. patent on this admixture was granted to the Army on 22 March 1994. The low-temperature strength performance of KC1 was significantly superior to all other admixtures tested. However, KC1 contains significant amounts of sodium, and therefore may pose a durability concern if used with alkali-reactive aggregates.

The research team met at the end of each task to discuss the test results and future directions. Some of the admixtures tested in early tasks were then reformulated, and other new formulations were included in later tasks. The formulations were chiefly based on predictions of the freeze-point depression and strength acceleration effects of individual compounds.

Because portland cement is a mixture of various chemical compounds that have individual chemical properties, and the admixtures were made of more than one chemical compound, the combined chemical system for each concrete mix was very complex. The research process involved several cycles of formulation and empirical validation. Therefore, although some tasks did not produce satisfactory admixtures, they provided a foundation for the development of better admixtures in later tasks. For example, some admixtures yielded good low-temperature strength, but set too quickly to allow proper transport, placement, and consolidation. Other admixtures kept concrete from developing ice, but did not produce adequate strength.

At the end of each task, the research team examined the test results, improved the admixture formulations, and planned the next task. The process included ten tasks until satisfactory admixtures were produced. Although the chief selection parameter for the admixtures of Phase 1 was their strength performance at –5°C at seven days, other important parameters considered were alkali content, strength performance at room temperature, corrosion potential, workability, slump loss, and cost.

Two admixtures were selected for more comprehensive evaluation in Phase 2. These showed good strength enhancement at low temperature and did not contain significant alkalis or chloride ions.

Phase 2: Laboratory evaluation of best admixtures from Phase 1

Objective

To evaluate the most promising admixtures from Phase 1 in terms of the parameters most relevant to their use in concrete. (See Table 6.)

Table 6. Tasks included in Phase 2.

Task no.	*Title*
1	Set times
2	Corrosion
3	Hydration products
4	Alkali-silica reaction
5	Air-void analysis
6	Shrinkage

Experimental approach

ASTM C 494 contains standards for chemical admixtures used at temperatures above freezing. There is no specific standard for admixtures below the freezing point of water. The parameters chosen for the evaluations in Phase 2 were selected to approximate the standards provided in ASTM C 494 to the extent possible. Two antifreeze admixtures, code-named DPTC and DP, were selected based on their strength performance at –5°C. Daraset did not perform as well as these admixtures at –5°C, and therefore was not tested further. Daraset performs well at temperatures above freezing. PolarSet is a new admixture de-

veloped by WRG and sold as a low-temperature set accelerator. PolarSet was tested in Task 1F, but its strength performance at –5°C did not meet the preset acceptance criteria. Neither DP nor DPTC met the minimum strength requirements at –5°C. PolarSet was carried into Phase 2 as a reference and became a commercial product during the course of this project.

Concrete containing the selected admixtures and control concrete was mixed, cast at room temperature, and then cured at –5°C. Specimens from this concrete were subjected to a series of laboratory tests geared to characterize specific properties.

Phase 2, Tasks 1 to 6

This phase was divided into six tasks, each testing one relevant parameter. In addition to admixtures, the concrete used in the Phase 2 tests was made with the mix proportions shown in Table 7.

Task 1: Set times. In practice, workers must mix, place, and consolidate concrete within a limited time because concrete gradually changes from a viscous paste to a rigid material. Therefore, it is important that concrete be placeable and remain workable for a reasonable time after mixing. The loss of slump with time, and the setting times as defined in ASTM C 143 and ASTM C 403, respectively, were measured at room temperature on mixes containing the selected admixtures.

Table 7. Concrete mix design for specimens tested in Phase 2.

Cement type	Portland Type I
Cement factor	418.6 kg/m^3
Coarse aggregate	1102 kg/m^3
Sand	583.1 kg/m^3
Water	174.6 kg/m^3

Two sets of mixtures labeled "A" and "B" were tested. They were labeled this way to relate mixes that have the same antifreeze admixture and dosage. Table 8 shows that, with proper dosage of the admixtures, the slump and air content can be controlled. These mixtures had slumps of 15 cm ± 2 cm, and air contents of 6% ± 1%. Table 8 also shows the setting times for each mixture. By comparing these setting times to the control concrete, the effect of each admixture on the setting times can be observed. It is interesting to compare the setting times of the candidate antifreeze admixtures to those of PolarSet, which is already an accepted commercial admixture.

Except for DPTC at 6%, all setting times were increased by the antifreeze admixtures. Previous studies by WRG have found that relatively high

Table 8. Set times.

Mix no.	*Antifreeze admixture and dosage (% s/s)**	*WRDA-19† (g/kg)***	*Daravair†† (g/kg)***	*Slump (in.)*	*Fresh concrete air (%)*	*Initial set (hr)*	*Final set (hr)*
1A	Control (no antifreeze admixture)	5.0	0.8	13	6.0	4.8	6.6
2A	PolarSet, 6%	7.8	0.6	17	6.6	4.1	11.9
3A	DPTC, 6%	7.6	0.6	17	5.9	3.8	9.4
4A	DP, 6%	7.5	0.7	17	5.8	5.8	13.6
5A	PolarSet, 8%	8.1	0.6	13	6.0	8.2	N/A
6A	DPTC, 8%	4.1	0.5	13	5.6	>9	N/A
7A	DP, 8%	8.1	0.6	16	5.9	>9	N/A
1B	Control (no antifreeze admixture)	5.0	0.8	15	5.9	4.8	7.0
2B	PolarSet, 6%	7.7	0.8	15	6.9	8.0	12.2
3B	DPTC, 6%	9.2	0.9	13	5.3	5.3	10.6
4B	DP, 6%	9.2	0.7	15	5.5	7.6	11.9
5B	PolarSet, 8%	10.2	1.1	13	6.2	6.3	14.0
6B	DPTC, 8%	10.2	1.2	13	6.7	8.9	N/A
7B	DP, 8%	10.2	1.2	14	7.0	>9	N/A

* % s/s = percent of weight of the solids in the antifreeze admixture by weight of cement.
† WRDA-19 is a commercial water-reducing admixture.
** g/kg = Amount of admixture in grams of admixture per kilogram of cement.
†† Daravair is a commercial air-entraining admixture.
N/A = Data not available

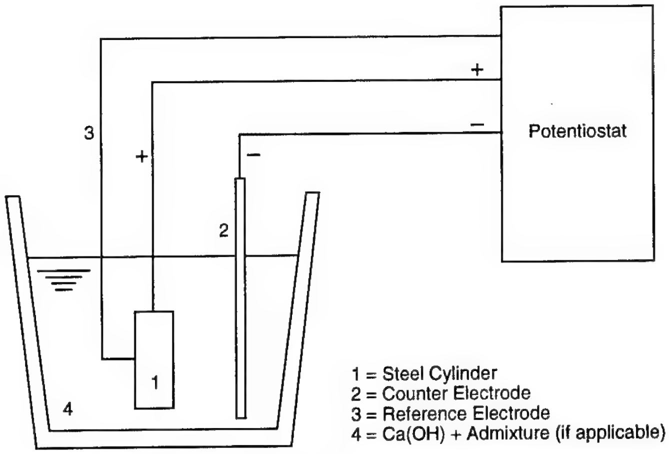


Figure 2. Cyclic polarization test.

dosages of set accelerators (i.e., > 6% s/s) formulated with various calcium salts can prolong the set of concrete compared to a control mix. In practice, set accelerators are rarely used above 4% s/s. The set times for the mixtures containing DP or DPTC at 6% are similar to those of the mixture containing PolarSet.

Task 2: Corrosion. Two types of tests, 1) cyclic polarization test, and 2) lollipop test, were conducted.

1) Cyclic polarization test. The most promising chemical admixtures (code names DP and DPTC) and a control were evaluated for their tendency to pit by a cyclic polarization test. This test subjects the surface of a metal to conditions promoting pitting in environments similar to those found in concrete pore water. A 9-mm-diameter, 13-mm-long steel cylinder is immersed in a saturated calcium hydroxide solution with and without sodium chloride (Fig. 2). The steel cylinder is polarized from –800 mV versus saturated calomel electrode (SCE) at a scan rate of 5 mV/s until the current reaches 255 $\mu A/cm^2$, at which point the potential is reversed. The test ends at a potential of –700 mV versus SCE.

The test determines the pitting tendency of admixtures. Three important data points are obtained:

E_b = breakdown potential (the potential at which pitting starts).
E_p = pitting potential (the potential below which pitting cannot occur).
I = current density 200 mV below the pitting potential.

The more negative the values of E_b and E_p, the less effective is the admixture as a corrosion inhibitor. The magnitude of the current density at 200 mV lower than the pitting potential gives an indication of possible cathodic inhibition. The test results for two specimens for each condition are shown in Table 9. The full test charts are also included for reference for those familiar with these electrochemical tests. Specimens 1A and 1B were the control specimens (no antifreeze admixture) tested in a saturated calcium hydroxide solution also containing sodium chloride at 5% by weight of water, with no other concrete admixture present. Notice that these specimens showed the most negative pitting potential (more prone to pit), as expected. Specimens 2A and 2B were like 1A and 1B, except that the antifreeze admixture code-named DP was included in the solution at 6% by weight of water. Notice that the pitting potential was much more positive, which indicates less pitting tendency with this antifreeze admixture.

2) Lollipop test. This test measures the corrosion that can occur in a steel rebar partially embedded in a concrete cylinder. The rebar protrudes from the concrete cylinder, which is partially immersed along its longitudinal axis to half its height in a 3% sodium chloride solution. The initial resistivity is measured using standard AC impedance techniques.

Six specimens for each admixture and a control solution were tested. The test results are shown in Table 10. The data suggest that these admixtures reduce the corrosion rate compared to the control specimens. Therefore, from the standpoint of corrosion potential, these admixtures do not lead to increased corrosion of embedded steel.

Task 3: Hydration products. Strength, durability, and other concrete properties are affected by the composition and microstructure of the products formed during hydration. The composition, structure, and overall quality of a hardened cement paste are determined primarily by four factors: 1) type and amount of cement, 2) the water/cementitious material ratio, 3) moisture availabil-

Table 9. Pitting potential.

Test no.	*Solution [in addition to H_2O and $Ca(OH)_2$]*	*Pitting potential (mV)*
1A	NaCl	–550
1B	NaCl	–525
2A	DP + NaCl	–29
2B	DP + NaCl	–39
3A	DPTC + NaCl	–59
3B	DPTC + NaCl	–80

Table 10. Corrosion rates with various admixtures.

Sample no.	*Admixture*	*Initial resistivity (kohms/cm)*	*2-year corrosion potential (mV vs. SCE)*	*Corrosion rate $\mu A/cm^2$ (relative)*
1A	Control	5.4	–249	0.23
1B	"	5.3	–564	0.70
1C	"	5.5	–536	0.28
1D	"	6.1	–91	0.05
1E	"	5.7	–211	0.11
1F	"	5.5	–131	0.08
Average	"	5.6	–297	0.24
2A	PolarSet*	3.4	–86	0.06
2B	"	3.8	–94	0.07
2C	"	3.2	–65	0.06
2D	"	3.2	–76	0.06
2E	"	3.8	–80	0.09
2F	"	4.0	–75	0.05
Average	"	3.6	–79	0.07
3A	DP*	5.2	–108	0.07
3B	"	4.6	–86	0.02
3C	"	4.5	–121	0.05
3D	"	4.8	–136	0.07
3E	"	4.3	–97	0.06
3F	"	4.8	–73	0.05
Average	"	4.8	–104	0.05
4A	DPTC*	4.4	–114	0.06
4B	"	5.1	–119	0.12
4C	"	5.3	–127	0.08
4D	"	4.5	–146	0.18
4E	"	4.6	–113	0.19
4F	"	4.6	–118	0.17
Average	"	4.8	–123	0.13

*The given admixture was dosed at 6% of solids by weight of the water in the solution.

ity and curing temperature, and 4) the type and amount of admixtures. A detailed microscopic examination of concrete specimens containing selected admixtures can provide insight into the effects that such admixtures can have on the compressive strength and long-term durability of hardened concrete.

The evaluation and characterization of the concrete microstructure included both conventional and advanced imaging techniques. Preliminary observations were conducted at relatively low magnifications (< 75×) on both fractured and polished surfaces, using a stereomicroscope. More detailed and advanced imaging involved the use of high magnification (100×–1000×) reflected and transmitted light microscopy. The latter technique was augmented by the use of ultrathin (< 30 µm) sections of the hardened concrete. Finally, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDXS) were used to analyze the hydration products in each specimen.

The results of a detailed microscopic examination did not reveal any evidence of unusual or unique microstructures in the specimens containing the antifreeze admixtures, as compared to paste structures observed in control specimens. However, the specimens containing DP at 6%, and those containing DPTC at 6% and at 8%, exhibited abnormally high concentrations of calcium hydroxide on exterior surfaces and interior aggregate sockets. The abundance of calcium hydroxide suggests that significant bleeding may have occurred. The control specimens, which contained only water reducer and air entraining admixtures, exhibited heavy concentrations of air-void clusters along the paste/aggregate bond interface. The mix design parameters (such as water/cementitious material ratio, aggregates quantities and qualities, and cement factor) were kept constant for all mixes. The admixtures used are listed in Table 11. Other than the differences noted above, the cement paste/aggregate bond interface did not show any difference in composition or structure from those of the control specimens.

Task 4: Alkali-silica reaction. The selected admixtures do not add sodium or potassium (the potentially harmful alkalis) to the concrete mix. Therefore, testing for alkali-silica reaction as planned was found to be unnecessary, and was cut from the testing program.

Task 5: Air-void analysis. The air content and the spacing factor are the main parameters that determine to a large degree the freeze–thaw durability of concrete. In this task, the total air content of each mixture series was measured in both the fresh and hardened states. The spacing factor, the average chord length, the number of voids per inch, the specific surface, and the paste content were determined on hardened concrete specimens representing each of four mixtures. An air entraining agent, Daravair, was dosed to produce an air content of 5–7%. Also, a super plasticizer, WRDA-19 (Daracem 19), was dosed to produce a target slump between 13 and 17 cm. The water/cementitious material ratio was 0.417. The air-void structure in the hardened concrete was analyzed after 28 days of curing. The control specimens were cured at normal room temperature, while all of the antifreeze test specimens were cured at –7°C. The specimens were polished slabs cut from concrete cylinders.

Table 11. Air content in fresh concrete.

Mix no.	Antifreeze admixture and dosage (% s/s)*	WRDA-19 (g/kg)†	Daravair (g/kg)†	Slump (cm)	Curing temperature air (°C)	Fresh concrete (%)
1A	Control (no antifreeze admixture)	5.0	0.8	13	22	6.0
2A	PolarSet, 6%	7.8	0.6	17	−7	6.6
3A	DPTC, 6%	7.6	0.6	17	−7	5.9
4A	DP, 6%	7.5	0.7	17	−7	5.8
5A	PolarSet, 8%	8.1	0.6	13	−7	6.0
6A	DPTC, 8%	4.4	0.5	13	−7	5.6
7A	DP, 8%	8.1	0.6	16	−7	5.9
1B	Control (no antifreeze admixture)	5.0	0.8	15	22	5.9
2B	PolarSet, 6%	7.7	0.8	15	−7	6.9
3B	DPTC, 6%	9.2	0.9	13	−7	5.3
4B	DP, 6%	9.2	0.7	5.75	−7	5.5
5B	PolarSet, 8%	10.2	1.1	15	−7	6.2
6B	DPTC, 8%	10.2	1.2	13	−7	6.7
7B	DP, 8%	10.2	1.2	14	−7	7.0

* % s/s = Percent of weight of the solids in the antifreeze admixture by weight of cement.
† g/kg = Amount of admixture in grams of admixture per kilogram of cement.

1) Fresh concrete air content. The air content of each mix was measured within eight minutes from the end of the mixing. Table 11 shows that the target air content and slump were achieved with and without the antifreeze admixtures. Therefore, these admixtures are compatible with the water reducer and the air entraining admixtures tested, and caused no detrimental effect.

2) Hardened concrete air content. The total air content and parameters of the air-void systems were measured on concrete slabs, which were cut from the center of cylinders cast from each of the previously described mixtures. Each cylinder size was 7.6 cm × 15.2 cm. The cylinder numbers correspond to the individual mixture identification, disregarding whether they came from groups A or B. Only the first four mixtures listed in Table 11 were analyzed. As indicated in that table, mixture 1 represents a control concrete with a super plasticizer and an air-entraining admixture, but with no antifreeze admixture. Mixture 2 contains the same ingredients as mixture 1, except for the commercial low-temperature admixture PolarSet dosed at 6% of solids by weight of portland cement. Similarly, mixture 3 contains the selected antifreeze admixture DPTC at 6% dosage. Mixture 4 contains the selected antifreeze admixture DP. The air contents and other air-void parameters are summarized in Table 12.

ACI 201 *Guide to Durable Concrete* and ASTM C 457 recommend that the total air content be between 4.5% and 7.5%, and that the specific surface be greater than 24 (1/mm), and that the average spacing factor be 200 µm or less. The test results indicate that mixtures 1 and 2 meet the required air content. Mixture 3 is only slightly short of meeting the minimum air content, while mixture 4 had an excess of air voids. Too much air content may weaken the strength of concrete. The total air content in mixture 3 is slightly (0.1%) below the recommended minimum value for durable concrete. However, its spacing factor is favorable, which indicates an abundance of very small voids that may provide adequate freeze–thaw protection.

The total air content and average void spacing factor are the most important parameters for gauging the freeze–thaw durability of hardened concrete. The use of antifreeze admixtures does

Table 12. Air-void parameters in concrete containing antifreeze admixtures.

Air-void parameters	Mixture 1	Mixture 2	Mixture 3	Mixture 4
Air content (%)	5.5	7.5	4.4	8.5
Chord length (µm)	124	135	109	152
Voids per cm	4.3	5.5	3.9	5.5
Specific surface (1/mm)	31.9	29.8	36.9	26.1
Spacing factor (µm)	152	137	145	140
Paste content (%)	30.7	30.7	30.7	30.7

Table 13. Shrinkage of concrete containing antifreeze admixtures.

Mixture no.	Admixture	Dosage (%)	Percent length change at 4 days	Percent length change at 7 days
1A	Control	0	–0.008	–0.024
1B	Control	6	–0.007	–0.025
2A	PolarSet	6	–0.020	–0.046
2B	PolarSet	6	–0.009	–0.031
3A	DPTC	6	–0.003	–0.022
3B	DPTC	6	–0.005	–0.023
4A	DP	6	–0.001	–0.019
4B	DP	6	–0.003	–0.016
5A	PolarSet	8	–0.014	–0.045
5B	PolarSet	8	–0.021	–0.052
6A	DPTC	8	–0.024	–0.054
6B	DPTC	8	–0.016	–0.049
7A	DP	8	–0.019	–0.049
7B	DP	8	–0.015	–0.045

* Negative sign indicates shrinkage (negative expansion).

not seem to have a significant effect on the overall quality of the air-void parameters. Therefore, it is deduced that these admixtures do not adversely affect the freeze–thaw durability of concrete.

Task 6: Shrinkage. In addition to strength and durability, concrete must exhibit adequate volume stability. Excessive expansion or contraction of concrete elements can lead to joint damage, bulging, and cracking. Chemical admixtures have been known to affect the drying shrinkage of concrete during the curing period. Concrete mixtures containing the candidate admixtures and admixture-free control mixtures were tested for drying shrinkage that occurred during their first seven days of curing. Beam specimens (25 mm^2, 285 mm in length) were cast and tested according to ASTM C 157. The specimens were mixed and cast at room temperature. The control specimens were cured at room temperature and the others were cured at –7°C. Their initial length was measured 24 hours after casting. The specimen lengths were measured again four and seven days later. These measurements were compared to the initial reading for each specimen. The length changes expressed as a percentage of the initial lengths for each specimen are presented in Table 13.

The data show that there was a significant increase in the shrinkage of all specimens containing the admixtures at the higher percent (8%). While PolarSet at 6% increased the shrinkage by more than 50% compared to the shrinkage of the control specimens, use at the normal dosage range recommended by the manufacturer conforms to the requirements in ASTM C 494. DPTC at 6% slightly decreased the shrinkage. DP at 6% decreased the shrinkage by 29% compared to the control specimens.

The shrinkage test results suggest that the candidate antifreeze admixtures are benign to concrete at 6% dosage. The 8% dosage shows a tendency toward increased shrinkage. The potential for shrinkage should be checked on actual job concrete when admixtures are used above 6% dosage.

Phase 3: Prototype slab-wall application

Objective

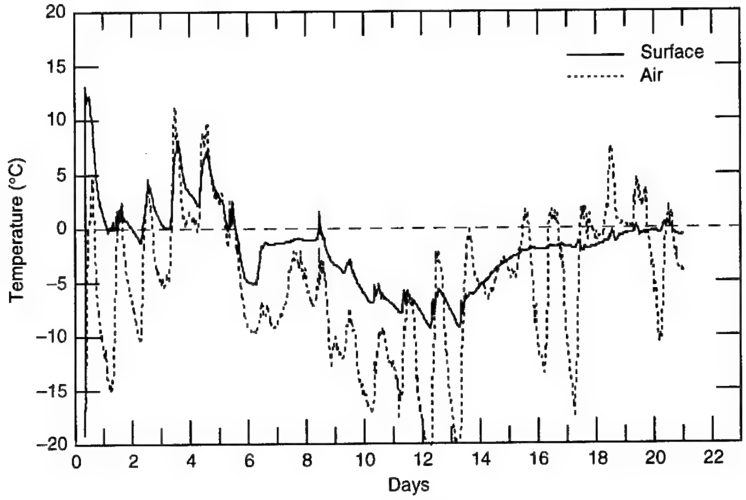
To assess the practicality of using the newly developed antifreeze admixtures in a full-scale structure with low risk and easy access for monitoring.

Experimental approach

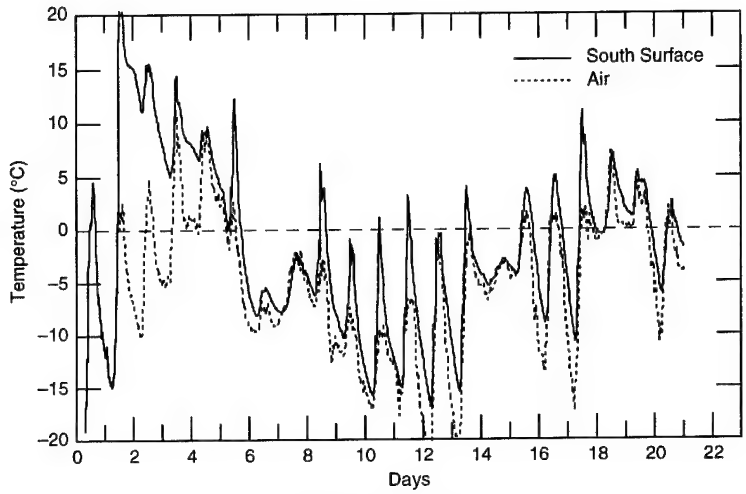
Numerous chemical combinations were investigated for approximately two years before two formulations were selected for final consideration. The two prototype admixtures are referred to as DP and DPTC. Disclosure of their chemical composition is withheld as the formulations are proprietary.

At CRREL, a composting bin consisting of a 16.5-cm-thick reinforced slab on grade 3.7 m wide by 4.6 m long with 0.9-m-high, reinforced 20.3-cm-thick walls on three sides was cast 17–18 February 1994. Site preparation consisted of removing a meter of snow from the ground, placing about 10 cm of dry sand on the newly exposed but frozen ground, and setting the forms and reinforcing steel. The concrete was placed in the forms, consolidated, and finished as usual. A plastic sheet was placed over the concrete for three days to minimize water loss. The wood forms were removed from the walls 20 hours after the concrete was cast. No thermal protection was provided to the concrete. Plastic pullout cylinders 10 cm in diameter by 15 cm long were cast into the slab and the top of the wall to provide in-situ strength-gain results. No control concrete was cast on this application.

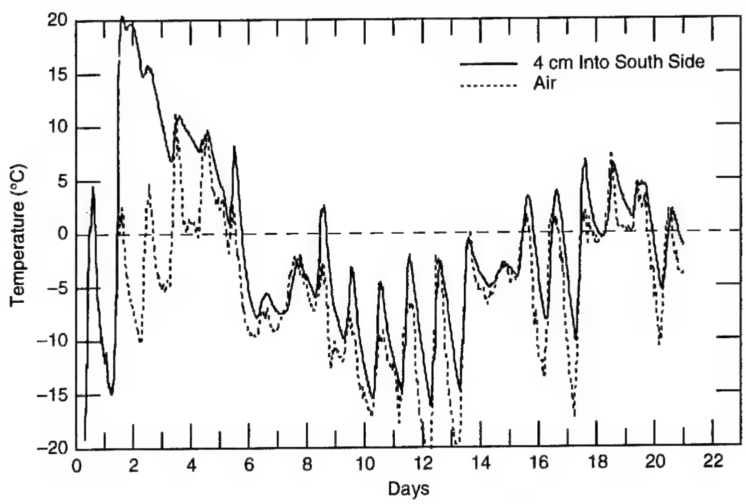
Thermocouples were embedded in the concrete at selected locations to monitor the air and concrete temperatures by means of electronic data loggers. Special attention was given to workability, finishability, thermal effect of the concrete mass, temperature gradients, and strength development.



a. A slab surface.



b. A wall surface.



c. A point 4 cm into wall.

Figure 3. Thermal history of various surfaces.

Observations and results

Workability

The concrete water content was reduced at the ready-mix plant to account for the water to be added with the admixtures to the concrete truck at the construction site. Accordingly, the water/cement ratio at the plant was 0.34 and became 0.43 after the respective admixture solution was added. The negative effect of holding back water during initial mixing was that the concrete air contents were very low (i.e., 3% instead of the desired 6%). The impact of such a low air content is that the concrete may not be as durable as it would have been had the concrete contained 6% air. It was also noticed that adding the admixture at the mix plant, though producing a concrete of correct air content, had the tendency to result in stiffer mixes at the job site. The DPTC mixture lost slump relatively fast, perhaps because the concrete and the air temperatures were higher than anticipated, and because it did not have a plasticizer. The slump was 5 cm during placing.

Finishability

Finishing antifreeze concrete was the same as finishing normal concrete. The trowel did not freeze to the surface of the concrete and the resulting finish looked good. The main complaint from the finishers was that some of the mixes arrived very stiff. This may be corrected by adding a plasticizer.

Thermal record

Five thermocouples were equally positioned through the thickness of the slab and six through the wall beginning at the outside surface. An additional thermocouple was positioned slightly away from the concrete, out of direct sunlight, to record air temperature. The complete thermal records can be found in Appendix B. A 21-day temperature history for the most relevant locations is presented in Figures 3a–c. The outside surface was the coolest portion of the slab and wall. It cooled more quickly and experienced wider temperature excursions than the interior concrete.

Table 14. Concrete placement time.

Mix	*Date*	*Start*
PolarSet	17 Feb	9 a.m.
DP	18 Feb	11:35 a.m.
DPTC	18 Feb	11:55 a.m.

Table 14 shows the approximate time each concrete was placed. The air temperature during transportation of the concrete on the 17th began at –16°C, rose to a high of 4.5°C at 2:00 p.m., and then dropped off to well below freezing that night. The slab concrete temperature at placement was 10°C. It rose to 12°C by noon and then dropped off to –0.4°C by 4:00 a.m. Though the air temperature during the next three nights got quite cold (–15°C at 6:30 a.m. on the 18th, –10.3°C at 6 a.m. on the 19th, and –5.4°C at 2 a.m. on the 20th), the concrete did not freeze. It dipped to a low of –1.2°C, which is not a freezing temperature for these mixes, at 6:30 a.m. on the 19th. The slab finally cooled to below –5°C at 8:00 p.m. on the 26th, and remained below that temperature for five days, until 5 a.m. on 3 March. It then rose slowly for the next seven days to near 0°C on 10 March. These low temperatures, though harmful to fresh concrete, were not harmful to the nine-day-old concrete.

The low ground temperature, which acted as a heat sink, caused concern that the bottom of the concrete would not be able to warm the ground above –5°C , and that the concrete would freeze from the bottom up. The temperature data show that freezing did not happen. For several days the bottom of the concrete slab remained near 0°C. It remained slightly warmer than the top surface of the slab, even after a week of curing. This result has implications for normal winter concreting, in which placing fresh concrete on frozen ground is prohibited because of the danger of freezing.

The wall was placed on 18 February. The wooden forms were erected and the rebar was set during the morning. Concrete placement began at 11:35 a.m. Figure 3b gives the wall surface temperature history. The air temperature at 11:35 was 1.8°C, rose to 2.5°C at 3 p.m., and fell to below freezing that night. The concrete arrived somewhat warmer than the prior day's concrete. It began at 13°C, rose to 19.7°C at 3:30 p.m. and (unlike the slab) its coldest portion, the surface, did not cool off appreciably over the next several days. The combination of the insulation effect provided by the wooden forms and the fact that there was no cold substrate to place concrete against helped the concrete to remain warmer longer. The forms were removed at 9:30 a.m. on the 19th, allowing the concrete to cool somewhat but to remain significantly above ambient temperature until the 22nd, three days after being cast. From that point on, the wall temperature tracked air temperature, which indicates that much of the chemical reac-

Table 15. Mixture proportions per cubic meter, New Hampshire.

Mixture	Coarse aggregate (19-mm crushed ledge, 0.5% 2.89 SG) (kg)	Sand (natural, 1.1% abs. 2.71 SG) (kg)	Portland cement Type II (kg)	w/c ratio	Air-entraining admixture (Daravair) (cm^3)	Water reducer (WRDA w/Hycol) (cm^3)	Admixture dosage (wgt active ingredient per cement wgt) (%)
PS	1010	787	422	0.44	133	798	6
DP	1015	784	416	0.43	266	798	6
DPTC	1021	784	422	0.42	237	828	6

Table 16. Properties of fresh concrete, New Hampshire.

Mixture	Slump (cm)	Air content (%)	Unit weight (kg/m^3)	Concrete temperature (°C)
PolarSet	8	3	2293	10
DP	11	7.2	2293	13
DPTC	5	6.4	2341	13

tion between the cement and water had been completed by then.

The slab reached a mostly uniform temperature within 18 hours. The wall achieved uniform temperature almost immediately. The wall, having two surfaces exposed to the ambient air, was most easily affected by surrounding air.

Strength development

The concrete was transported by rotary-drum truck from a ready-mix plant 16 kilometers from CRREL. The concrete was mixed using unheated aggregate with heated water (82°C). The ingredients were added into the truck's drum, mixed a few minutes, and then transported 15–20 minutes to the construction site. The mix proportions are given in Table 15. The truck took about 15 minutes to discharge its load, and finishing operations took another 30 minutes. Table 16 gives properties of each mixture as delivered to the site.

Results of the strength tests from the field-cured pullout cylinders taken out of each concrete section are presented in Figure 4. The target slump was 10 cm.

Control, admixture-free concrete was not mixed during this experiment. The control curves included in Figure 4 correspond to concrete of similar mix proportions made with aggregates and cement from the same sources. These values were derived from previous laboratory data. As can be seen, all field samples performed well. They exceeded minimum strength requirements. In addition, the concrete was estimated to have attained at least 12 MPa before it reached –5°C. The concrete was able to resist freezing at that strength, shown by the fact that no ice damage was detected in core samples removed from the bin in May.

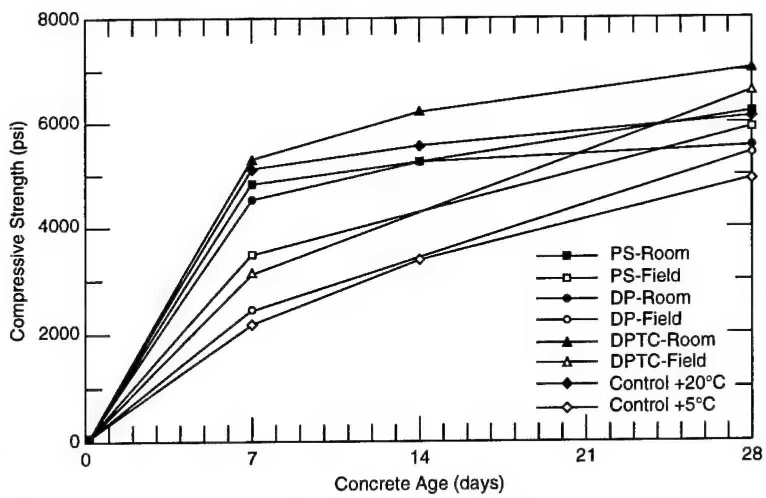


Figure 4. Compressive strength of pullout cylinders from wall-slab prototype.

FIELD APPLICATION OF DEVELOPED ADMIXTURES

Soo Locks slabs

Objective

To demonstrate the practicality of using antifreeze admixtures under field conditions.

Description

Three on-grade slabs were cast. One slab was made of admixture-free concrete to act as a control. It was protected with a heated shelter. A second slab was made with concrete containing the

antifreeze admixture code-named DP, which was developed in this project. A third slab contained the admixture PolarSet (PS), a low-temperature accelerator newly marketed by WRG. The admixtured slabs were cast and cured in the open without shelter. The prototype application in New Hampshire provided some experience in dealing with these admixtures in cold weather. The slabs at the Soo Locks provided an opportunity to apply the new admixtures in a realistic cold weather setting with conventional equipment and conventionally trained labor.

Each reinforced slab on grade measured 5.5 m wide by 6.1 m long by 15.2 cm thick. The slabs were cast 15–17 March 1994. The Corps' Soo Area Office had scheduled 39 sections of concrete to be replaced because of their advanced stage of freeze–thaw deterioration. The work area was located on the southwest pier, which borders the ship canal of the Soo Lock, the largest of four locks operated and maintained by the Corps of Engineers, Sault Sainte Marie, Michigan. Inspection and repair of the locks themselves is normally done during the winter months, January through March, when shipping is stopped. Other repair work, such as the replacement of the slabs described here, is also most conveniently done during the winter nonshipping season, making this test particularly relevant.

The temporary heated enclosure erected over the control slab provided a comparison between conventional and antifreeze concrete operations. The two admixtured slabs were placed on a cold gravel bed. After consolidation and finishing operations were conducted conventionally, the fresh slabs were covered with a plastic sheet. The concrete was exposed to ambient air and was in direct contact with the cold gravel base course, which was placed directly on frozen ground. No insulation, shelter, or heater was used on the admixtured slabs. The plastic sheet was placed over the two exposed concrete sections for seven days to minimize water loss because no curing water was added. The concrete in the heated shelter was left uncovered for the seven-day curing period.

Observations and results

Workability

The concrete stayed workable longer than it did in the prototype application in New Hampshire. The mixtures were 3–6°C colder in Michigan.

Finishability

The concrete workers indicated that the two mixes, DP and PolarSet, finished quite easily. The DP did seem to stiffen right at the end of the finishing operation, about two hours after water was first added to the mix. However, the concrete that was left over in a wheelbarrow from the sample-making procedure was still workable. The DP contained a different water reducer than that used in New Hampshire; WRDA-19 was used in New Hampshire, while AA1D was used in Michigan. The first is a commercial product of WRG while the second was still in research there.

Thermal records

Four thermocouples were equally positioned through the thickness of the PolarSet and DP slabs at about 2.5 m from the edge. One thermocouple monitored air temperatures.

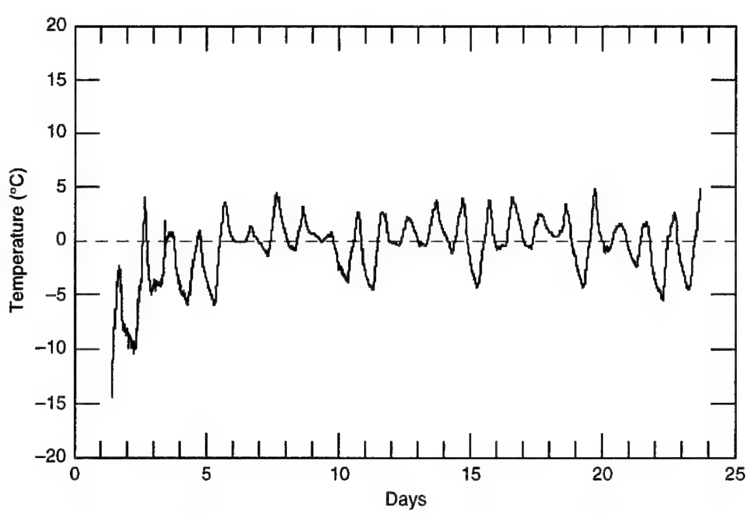


Figure 5. Air temperatures at the construction site.

Figure 5 shows a 22-day record of air temperature beginning at 9:50 a.m., 16 March. The air temperatures from 16–17 March were quite cold, averaging –10°C, with a low of –16.5°C at 6:45 a.m. on the 17th. Steady winds created wind chills of –28°C. The air temperature averaged only –0.5°C for the next seven days. The overall average air temperature for the first nine days was –1.6°C.

The control concrete was cast on 15 March. This slab was cast in a shelter that was heated for the first seven days; the heater was then turned off, but the shelter stayed in place for about a month. Nineteen days of temperature records from four positions

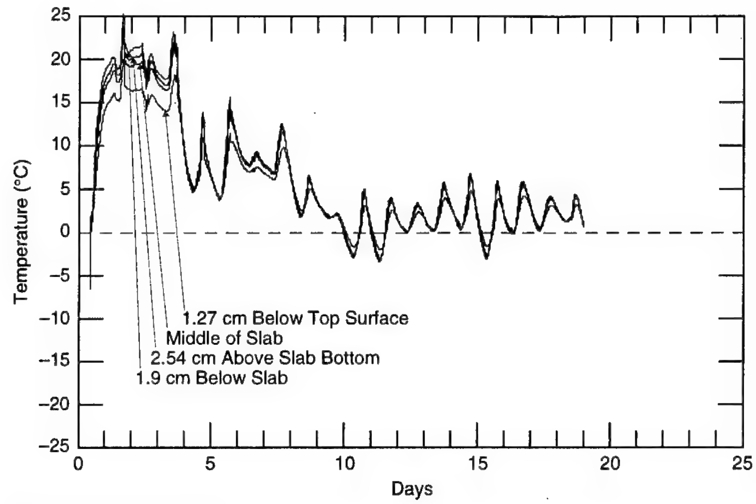


Figure 6. Concrete temperatures at various depths for the heated control slab.

through the thickness of the slab are given in Figure 6. The position 1.9 cm below the bottom of the slab was colder than the slab itself the first 2 1/2 days. Over that same 2-1/2-day period, the top surface was slightly warmer than the middle, which was slightly warmer than the bottom of the slab. Thereafter all temperatures throughout the slab were essentially the same. The heat was turned off in the control shelter by midafternoon on 22 March. By 10 a.m. on the 23rd, the control concrete had equilibrated to ambient conditions.

The PolarSet and DP sections were cast on 17 March. A malfunction in the data recorder prevented temperatures from being recorded past midnight, 18 March. Consequently, a two-day temperature record of four positions through the thickness of each slab is given in Figures 7 and 8. Both mixes were relatively warm during this short period, averaging 6.7°C and 6.3°C for the PS and DP, respectively. Neither mix is expected to have dropped below –5°C through 23 March. By then it would have been able to resist frost damage. Except for the heat of day when the sun was shining, each slab was of uniform temperature throughout its thickness. Based on tests conducted previously, it is expected that the temperature of the PS and the DP slabs tracked that of the air temperature after day 4. The surface may have gotten a few degrees warmer than the air during midday.

Microscopic evaluation of the hardened concrete

Core samples were cut from both slabs in July 1994 and examined for evidence of ice crystal imprints. No icing was found. Similarly, no ice was found in the 7.6- × 15-cm core samples that were

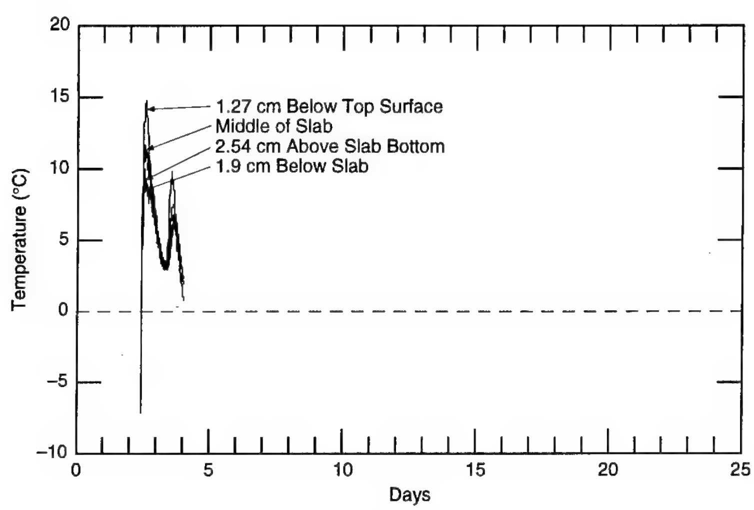


Figure 7. Concrete temperatures for the slab containing PolarSet.

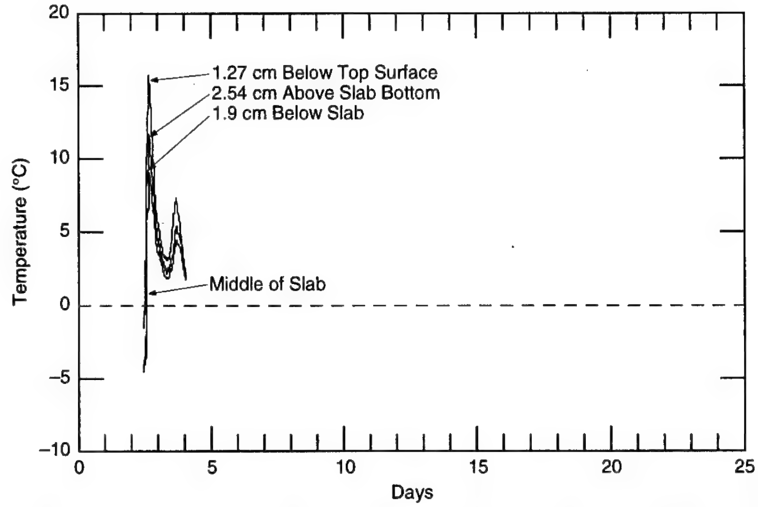


Figure 8. Concrete temperatures for the slab containing admixture DP.

stored unprotected next to the slabs. Air-void parameters were not determined.

Strength development

Concrete cylinders were cast during construction. For the heated control slab, the cylinders were stored for curing on the ground near the slab and on an overhead shelf. For the admixtured concrete slabs, the cylinders were stored for curing partially embedded in cold gravel at the edge of the concrete slabs. Also, a second set of cylinders for each admixtured concrete was stored on an overhead shelf in the heated enclosure. The cylinders were 7.5 cm in diameter by 15 cm in length. Figure 9 shows their compressive strengths at various ages. The strengths of the concrete cylinders containing the admixtures, stored heated and unheated, exceeded the strength of 28-day control concrete cured under the heated condition. The mixture proportions are given in Table 17, placement time of the concrete in Table 18, and the properties of fresh concrete in Table 19.

Cost comparison between conventional and antifreeze concrete

As previously mentioned, a heated shelter was used for the control concrete. This provided an opportunity to compare costs between normal winter concreting to those using antifreeze admixtures. Based on these field tests, it is apparent that the main difference between normal concrete and antifreeze concrete is the heat, shelter, and labor needed to protect normal concrete compared to the chemicals needed to protect antifreeze concrete. The cost to erect, heat, and dismantle the temporary shelter at Soo was estimated to be

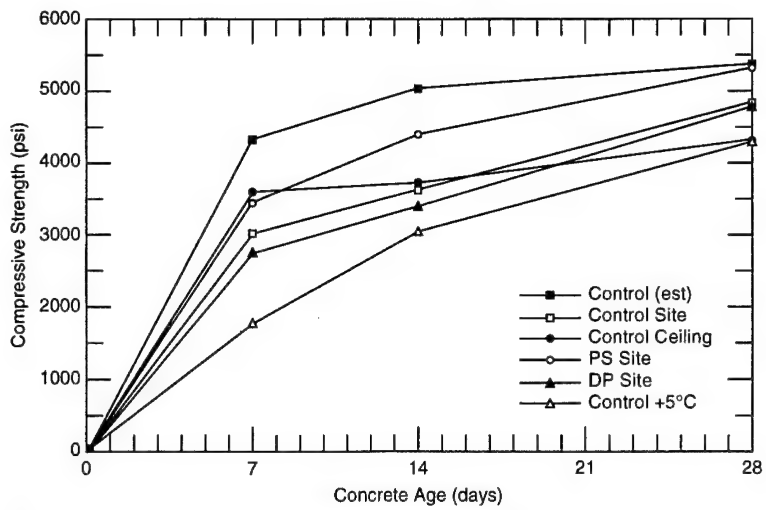


Figure 9. Compressive strength of cylinders (some cured in the heated shelter and some next to open slabs).

Table 17. Mixture proportions per cubic meter, Michigan.

Mixture	*Coarse aggregate (19-mm) (kg)*	*Sand (kg)*	*Portland cement Type IA (kg)*	*w/c ratio*	*Admixture dosage (wgt active ingredient per cement wgt) (%)*
Control	1045	772	391	0.41	0
PolarSet	1045	772	391	0.43	5.2
DP	1045	772	391	0.41	5.2

Table 18. Concrete placement time, Michigan.

Mixture	*Date*	*Start*
Control	15 Mar	11:00 am
PolarSet	17 Mar	10:50 am
DP	17 Mar	1:10 pm

Table 19. Properties of fresh concrete, Michigan.

Mixture	*Slump (cm)*	*Air content (%)*	*Unit weight (kg/m^3)*	*Concrete temperature (°C)*
Control	5	3.2	2309	12
PolarSet	11	5.3	2293	8
DP	13	8	2164	7

Table 20. Winter cost estimate.

Shelter	
Erect shelter (6 men, 1/2 day @ $23/hr)	$552.00
Heat shelter: 1 day prior to pour and 7 days after (8 days @ 81 L propane/day @ $0.206/L	$133.54
Dismantle shelter	$276.00
Materials: Assume 9 reuses (Total cost estimated at $1062)	118.00
Total estimated cost of shelter	$1079.54
Antifreeze admixture	
Volume of concrete placed inside shelter	5.12 m^3
Dosage of admixture per kg of cement	98 cm^3
Amount of cement per m^3 of concrete	391 kg
Amount of admixture per 5.12 m^3 of concrete	196 L
Cost of admixture to equal cost of shelter	$5.51/L

$1070. Heating accounted for nearly 15 percent of this expense. Because antifreeze admixtures are still prototypes, their market price has not been determined. However, based on the estimate developed for the shelter in this project (Table 20), the break-even cost of the antifreeze admixtures would be $5.51 per liter.

POTENTIAL APPLICATIONS OF ANTIFREEZE ADMIXTURES

The current standards were written under the assumption that concrete cannot develop strength at acceptable rates when the temperature is lower than 5°C. Current testing methods are designed for warm environments. Antifreeze admixtures open new possibilities and the challenge to adapt standards accordingly.

Antifreeze admixtures offer potential use in various applications. In building construction, floor slabs and wall sections can be placed without the need for temporary shelter. However, whether or not the method of antifreeze admixtures may be advantageous over conventional thermal protection depends on the specific job circumstances. For some jobs, building a construction shelter may be useful for worker comfort, at least during work periods.

Because of the ability to place concrete in the cold and let the concrete be cold while developing acceptable strengths, antifreeze mortar and concrete can be safely placed on cold substrates. This option allows for applications such as joints in precast concrete structures, repair of dams, tunnels, foundations, etc., where a massive structure is cold.

Winter paving operations can benefit greatly. With antifreeze admixtures, the concrete does not need to be thermally protected, and it can be placed directly on cold granular bases. From the standpoint of worker comfort, the highly mechanized operation of paving makes this type of work possible at even lower temperatures than for vertical building construction. Thus, there is ongoing need to search for admixtures that work at very low temperatures.

In general, summer construction is less expensive, but there are applications where the job must be done in the winter and against cold substrates.

There are other operations where work is best done during the winter when more workers are available and construction is less disruptive.

CONCLUSIONS

The experimental results of this project show that it is possible to place concrete at temperatures significantly below the freezing point of water and obtain acceptable structural strength. The test results also show that accelerating admixtures, such as Daraset, do not provide acceptable strengths in freezing temperatures. PolarSet, an accelerating admixture designed for cool temperatures, was developed and tested during this project. It performed very well at temperatures below 5°C, the current low-temperature limit for admixture-free concrete, provided the concrete was prevented from freezing. It was unable to provide protection down to the –5°C goal of this project. That required that a new admixture be developed.

A new prototype admixture code-named DP was developed in this project. DP was tested in the laboratory for compressive strength at various curing temperatures, and for several other relevant concrete properties. The laboratory tests indicate that this admixture met the pre-set requirement of causing concrete to gain strength at –5°C equal or higher than its equivalent, admixture-free concrete cured at 5°C. The tests also indicate that this admixture has no known detrimental side effects for concrete. It does not contain chlorides or alkalis, and therefore it does not induce corrosion or cause alkali-silica reaction. DP did not interfere with the function of an air-entraining agent known by the trade name Daravair.

Although DP needs to be tested prior to its use with other admixtures to ensure compatibility, so far there is no indication of compatibility problems. The strength of concrete containing DP and cured at 20°C was slightly enhanced compared to that of equivalent admixture-free concrete. DP was successfully applied to a winter field construction as described earlier. This field application indicated that concrete containing DP displayed adequate workability, adequate set regime, and adequate strength. The field application indicates that DP can be successfully applied without special equipment or specialized labor. The laboratory tests show that DP can be safely used with concrete cured at –5°C. This temperature is the internal temperature of concrete, not just the ambient temperature.

Caution must be used when comparing this temperature limit with the limits presented in the promotional literature of some commercial admixtures that present low-temperature limits of the ambient instead of the internal concrete temperature. In many cases, the ambient temperature may be below 0°C, but the internal concrete temperature may be above 0°C because of the heat of hydration and the presence of insulation. With those admixtures, the key to success is to keep the internal concrete temperature above freezing. With DP, the ambient temperature can be lower than –5°C, as long as the internal concrete temperature remains at about –5°C or higher. Neither Daraset nor PolarSet met the –5°C strength requirement that was preset at the beginning of this project. DP did. Daraset and PolarSet are excellent products for concrete temperatures above 0°C.*

Knowledge of the processes of ice formation in fresh and hardened concrete was advanced. Current winter practice assumes that concrete is automatically damaged if frozen before developing a compressive strength of 3.4 MPa. Experiments with antifreeze concrete showed that some ice in concrete does not necessarily cause damage, as long as certain minimum unfrozen water content is maintained. This added knowledge will be useful for future research in the areas of low temperature concrete and masonry. In the past, the temperature frontier for placing and curing concrete was at 5°C.

There are obstacles when introducing a new admixture to the market. The existing standards for concrete admixtures are not applicable to low-temperature admixtures because they evaluate the admixtures at higher temperatures. Not having industry standards discourages concrete practitioners from using a product because doing so would increase liability.

Admixture manufacturers must find a large-enough market to dilute the cost of promotion, storage, transportation, and distribution of a new admixture. Current winter concrete operations are relatively small because of the added cost of build-

*The manufacturer (Jeknavorian et al. 1994) has published information showing that concrete mixed with PolarSet at 22°C may not gain appreciable strength when cured at –5°C, but is able to regain strength when the concrete is warmed to 22°C. The 28-day strength for concrete cylinders treated in this manner is comparable to a control concrete cured at 22°C for 28 days.

ing with conventional methods according to existing specifications, and the lack of standards for the new low-temperature admixtures. Therefore, it becomes a somewhat negative cycle: users wait for commercially available admixtures and standards; admixture manufactures wait for the market to grow.

The U.S. Army Corps of Engineers is the largest civil engineering organization in the world. The Corps has provided leadership in many areas of civil engineering technology. The antifreeze admixtures technology presents both a challenge and an opportunity for the Corps to lead the way by developing the needed standards for working in the winter with concrete that is not heated. A proposal for changes to a Corps guide specification is included in Appendix C.

RECOMMENDATIONS

Daraset and PolarSet are two commercial admixtures that were evaluated during this project. Both of these are excellent admixtures for the range of temperatures for which they are specified. Neither of these admixtures met the minimum strength requirements set at the beginning of this project for concrete cured at –5°C. That necessitated the need to search for a new admixture. The admixture DP, developed in this project, met the preset requirements. Therefore, DP is recommended for use in winter concrete applications in which the internal temperature of concrete may be allowed to drop down to –5°C. The air temperature may be at even lower temperatures as long as the internal concrete temperature stays at –5°C. DP is not currently commercially available off the shelf. The investment needed to bring such a new admixture through the distribution channels, including storage tanks, advertising, technical advisor training, etc., is significant. At this point, WRG has decided not to make this investment until the winter concrete market grows. However, WRG is willing to supply this admixture upon request for significant projects.

The antifreeze admixture KC1 was patented by the U.S. Army. This admixture is made of two generic chemicals usually supplied in powder form. They can be purchased from any chemical supplier. KC1 is made of three weights of sodium nitrate (sodium nitrite works as well) and one weight of sodium sulfate. The recommended dosages vary from 6 to 8% by weight of cement. KC1's major disadvantage is that it adds alkalis to the concrete mix. This may pose a problem if reactive siliceous aggregates are used. Alkali-silica reaction is not an issue with calcareous aggregates such as limestone. Natural sand is made of siliceous mineral, but it is chemically inert, and therefore does not react with alkalis. Consequently, KC1 should be safe for use in mortar and grouts that have only natural sand as aggregate. For combat engineering applications and for emergency construction, where the short-term goals are predominant, KC1 would be recommended.

Much work is needed in promoting standards for antifreeze admixtures to encourage their demand. Admixture manufacturers must consider many factors in assessing the convenience of launching a new admixture into the market. Some of these factors are beyond technical control. Tort liability concerns discourage private construction industry in the United States from trying new materials for which industry standards are not available. Industry standards are difficult to set without extensive product application. Admixture producers would like to have the market demand before investing in a new product. The U.S. Army Corps of Engineers may be a catalyst for the implementation of this new technology. It is recommended that the Corps use the information in Appendix C to update its specifications. The information does not identify acceptable chemicals but, rather, identifies how they should perform when used at low temperature.

The return in federal research and development investment in a new technology, such as antifreeze admixtures, may be slow because of the standards, liability, and marketing considerations described above. However, the economic opportunity to increase the construction season in a significant portion of the United States is a real possibility that should not be ignored.

The U.S. Army could benefit from adding the antifreeze admixture method to its menu of options for winter operations. The experience in military construction could provide confidence in the use of antifreeze admixtures in the civilian sector.

TECHNOLOGY TRANSFER AND MARKETING PLAN

Objective

To disseminate and publicize the findings of the research project, and develop a marketing strategy as appropriate.

Technology transfer

Transfer of information is being achieved through the publication of technical reports, papers, and articles in professional journals, newsletters, and other engineering publications.

WRG has integrated the laboratory results and experience gained from this project into its technical and marketing literature.

Six publications were produced from this project (see the first six references in this report's "Literature cited" section).

Marketing plan

Market analysis

No comprehensive study of the market potential for freeze protection admixtures (also called antifreeze admixtures) has been carried out, but several pieces of information can be used to make a convincing case that a substantial market does exist. More detailed analysis is clearly required to more accurately define the opportunity for these kinds of admixtures.

To obtain an estimate of the current amount of concrete construction taking place under conditions that warrant freeze protection, monthly cement shipments on a state-by-state basis were matched with monthly average daily temperature data for the major markets within each state. Assumptions were then made concerning average amounts of cement per cubic meter of concrete, as well as the proportion of cement that goes to ready-mixed concrete construction.

Using this methodology for the 1992 calendar year, it is estimated that roughly 19 million cubic meters of ready-mixed concrete were placed in conditions where freezing conditions exist (approximately 10% of the U.S. total ready-mixed concrete). It is assumed from industry knowledge that only a small portion of this concrete was placed in environments that are heated for reasons other than providing adequate working/curing conditions. It is further estimated from company data that accelerators (both chloride and nonchloride) would have been used to treat anywhere from two to four million cubic meters of this total to provide accelerated set and strength development as well as some measure of freeze protection.

Based on these data and these assumptions, the statistics shown in Table 21 are derived. No work has been done as part of this project to survey the market and determine the amounts of money being spent on freeze protection. Previous estimates of $800 million per year for freeze protection have been published. The magnitude of this number matches well with the magnitude of the numbers presented in Table 21 ($600 million for current practice potential market using freeze-protection admixtures).

Economic viability of new admixtures

It is the opinion of the industry partner in this project that the products developed through this CPAR project definitely represent a positive advancement in the development of freeze-

Table 21. Market estimate.

Freeze protection available and potential market					
Available market		*Current practice Potential market*		*Future practice Potential market*	
(million m^3)	*($ million)*	*(million m^3)*	*($ million)*	*(million m^3)*	*($ million)*
2–4	22–38	15	600	29	1,140

Notes:

1. Available market is defined as the amount of concrete where admixtures are already being used to deliver some degree of freeze protection.
2. Average price of $9.81/m^3$ (to the concrete producer) for admixtures currently used in marginal freeze protection environments is assumed (dominated by inexpensive calcium chloride).
3. Current practice potential market merely assumes that all of the concrete currently protected from freezing through external or internal heating insulation could make use of an admixture to provide freeze protection.
4. Average price of $39/m^3$ is assumed for freeze protection of more than just a marginal nature.
5. Future practice potential market assumes that the availability of reliable freeze protection technology would make concrete construction during subfreezing weather more easily accomplished than it is now. The rate at which concrete is used where average daily temperatures are between 4°C and 16°C was applied for the amount of time that average daily temperatures are below 4°C to determine this potential. (This assumption resulted in a 90% increase versus current cement usage in freezing conditions.)

protection admixtures, also referred to as antifreeze admixtures. These new admixtures do not adversely affect the long-term durability of concrete. The admixtures developed through the joint efforts of WRG and CRREL provide satisfactory strength performance down to –5°C, exceeding the strength performance of untreated concrete cured at 5°C. There is limited use of chemical admixtures below 0°C. At any concrete curing temperature in excess of 0°C, PolarSet (WRG's new, premier nonchloride accelerator prescribed for air temperatures as low as –7°C) matches the strength of the new admixtures developed under the CPAR project. PolarSet also provides longer set times at its working temperature range. Therefore, the advantage band provided by the new antifreeze admixture is relatively narrow compared to PolarSet, and it may economically justify its use only for large projects where the job weather conditions can be fairly predicted.

Plans for commercialization

Bringing a new product to the market involves developing manufacturing facilities and protocols, implementing storage containers, increasing inventory costs, developing technical and promotional literature, and incurring training and other expenses. The advantage provided by the new product must justify the investment. In the case of the new admixtures developed under this CPAR program, the advantage was clear but not extensive enough to justify the cost of marketing a new product. The new admixture extends the concrete curing temperature approximately 5°C lower than that possible with WRG's existing accelerator, PolarSet.

The product as developed in this CPAR project will be further studied to see if it warrants commercialization on a very limited scale. It is currently anticipated that, if the product were to be brought through WRG's product authorization procedure, and then just kept on the shelf, it would be available on a specific project-by-project basis where its particular performance characteristics may be needed. Also, developments from this program are currently being investigated further to determine whether the performance of existing accelerator products can be enhanced. It is very difficult at this time to project future sales volumes for a product that is commercialized in this manner.

Given the magnitude of the market potential (even allowing for the cursory nature of the analysis), WRG views this as an exciting new opportunity for growth, and one that will be pursued further.

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APPENDIX A: PHASE 1 TEST RESULTS

Table A1. Compressive strength.. Task 1A cured at various temperatures.

a. 20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	4691	5293	5603
2	5955	6401	6923
3	6280	6106	6682
4	6813	6955	7026
5	5186	5870	6494
6	6130	6188	6791
7	7945	7827	8333
8	6342	6636	7533
9	4611	5186	5758
10	4385	4821	5857
11	5234	6130	6564
12	5568	6566	7432
13	4286	5057	5541
14	5021	5823	6060
15	5639	6318	6319
16	5677	6707	4803
17	4743	4951	5635
18	4677	5163	5588
19	4894	5352	5883
20	4583	5151	5494

b. –5°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	103	183	377
2	700	844	1160
3	1602	1973	1627
4	858	1317	2033
5	N/A	N/A	N/A
6	726	839	1028
7	1980	2053	2565
8	933	1200	1495
9	N/A	N/A	N/A
10	355	459	646
11	457	702	863
12	236	497	844
13	N/A	N/A	N/A
14	429	556	556
15	749	1296	1115
16	582	1075	2098
17	70	198	292
18	1589	1699	1926
19	3503	4347	5032
20	3489	4286	4838

c. –10°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	58	80	189
2	306	367	462
3	246	392	622
4	41	131	472
5	N/A	N/A	N/A
6	241	298	434
7	227	414	745
8	240	360	594
9	N/A	N/A	N/A
10	169	194	212
11	67	129	208
12	89	161	236
13	N/A	N/A	N/A
14	187	269	292
15	246	357	478
16	150	290	544
17	42	60	129
18	518	706	797
19	2072	2602	3065
20	2635	3791	3999

d. –20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	N/A	N/A	106
2	76	19	212
3	56	9	193
4	1	3	80
5	N/A	N/A	N/A
6	24	43	278
7	43	28	273
8	56	59	323
9	N/A	N/A	N/A
10	24	56	71
11	0	13	28
12	7	16	21
13	N/A	N/A	N/A
14	0	30	19
15	17	24	33
16	3	12	24
17	N/A	N/A	N/A
18	0	16	21
19	384	408	603
20	189	409	652

Table A2. Chemical composition of admixtures tested in Phase 1, Task A.

Mix no.	*Antifreeze admixture formulation*	*% Solids in sol.*	*Dosage (% CWT)*	*w/c Ratio*	*Slump (cm)*	*Remarks*
1	Control	0	0	0.48	5.7	
2	Daraset	30	2	0.48	N/A	
3	Daraset	30	3	0.48	N/A	
4	Daraset	30	4	0.48	N/A	
5	Control	0	0	0.48	2.5	
6	ACL	33	2	0.48	N/A	
7	ACL	33	4	0.48	N/A	
8	ACL	33	6	0.48	N/A	
9	Control	0	0	0.48	4.5	
10	X1B	50	2	0.48	N/A	
11	X1B	50	4	0.48	N/A	
12	X1B	50	6	0.48	N/A	
13	Control	0	0	0.48	5.7	
14	A-2	35	2	0.48	N/A	
15	A-2	35	4	0.48	N/A	
16	A-2	35	6	0.48	N/A	
17	Control	0	0	0.48	7.6	
18	KC1	25	2	0.48	N/A	
19	KC1	25	6	0.48	N/A	
20	KC1	25	8	0.48	N/A	

Table A3. Compressive strength. Task 1B cured at various temperatures.

a. 20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	4336	4810	5456
2	4687	4774	5461
3	4678	4822	5838
4	4357	4963	5352
5	4744	4805	5876
6	5112	5899	6107
7	6281	6550	6880
8	5725	5946	6486
9	4885	5340	6126
10	5470	6165	6909
11	5828	6719	7074
12	5390	5635	6842
13	5050	5570	6456
14	5102	5871	6593
15	5708	6166	6578
16	5913	6236	5956
17	4093	4774	5216
18	5083	6000	6611
19	5800	5965	6626
20	5607	6342	7008

b. –5°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	131	239	406
2	1339	1532	1608
3	3192	3923	4574
4	3040	4027	4574
5	N/A	N/A	N/A
6	1057	1448	1679
7	1415	2579	3103
8	967	1641	2325
9	N/A	N/A	N/A
10	726	1141	1344
11	1429	2551	2891
12	1325	2150	2089
13	N/A	N/A	N/A
14	1030	1141	1415
15	1841	3282	3824
16	1830	3183	3518
17	N/A	N/A	N/A
18	1806	2461	2938
19	2381	3674	4485
20	2164	3169	3933

Table A3 (cont'd).

c. –10°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	41	60	113
2	376	600	722
3	1386	2091	2363
4	1549	2457	2994
5	N/A	N/A	N/A
6	191	277	335
7	227	173	307
8	226	225	396
9	N/A	N/A	N/A
10	161	184	311
11	90	164	274
12	226	278	382
13	N/A	N/A	N/A
14	203	371	509
15	251	579	981
16	361	723	1269
17	N/A	N/A	N/A
18	287	401	472
19	213	546	1042
20	283	665	1070

d. –20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	*7*	*14*	*28*
1	0	0	71
2	45	66	170
3	260	535	877
4	254	677	1240
5	N/A	N/A	N/A
6	122	85	141
7	71	38	42
8	80	39	42
9	N/A	N/A	N/A
10	71	0	71
11	47	31	28
12	42	25	28
13	N/A	N/A	N/A
14	67	91	156
15	20	52	127
16	17	52	203
17	N/A	N/A	N/A
18	51	81	146
19	31	41	57
20	25	39	75

Table A4. Chemical composition of admixtures tested in Phase 1, Task B.

Mix no.	*Antifreeze admixture formulation*	*% Solids in sol.*	*Dosage (% CWT)*	*w/c Ratio*	*Slump (cm)*	*Remarks*
1	Control	0	0	0.48	5	Mix water at 9°C. Concrete temp at 23°C.
2	KC1	30	2	0.48	10	
3	KC1	30	6	0.48	11	
4	KC1	30	8	0.48	13	
5	Control	0	0	0.48	8	
6	CNN	35	2	0.48	6	
7	CNN	35	4	0.48	6	
8	CNN	35	6	0.48	4	Unworkable at 22 min.
9	Control	0	0	0.48	4	
10	CCT	33	2	0.48	4	
11	CCT	33	4	0.48	3	
12	CCT	33	6	0.48	3	
13	Control	0	0	0.48	8	
14	CCSD	35	2	0.48	5	
15	CCSD	35	4	0.48	5	
16	CCSD	35	6	0.48	3	
17	Control	0	0	0.48	3	
18	CCSN	33	2	0.48	3	
19	CCSN	33	4	0.48	4	
20	CCSN	33	6	0.48	3	

Table A5. Compressive strength. Task 1C cured at various temperatures.

a. 20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	4480	N/A	5659
2	4522	N/A	5324
3	4225	N/A	4989
4	4169	N/A	5060
5	4404	N/A	5725
6	4220	N/A	5036
7	4385	N/A	5347
8	4055	N/A	4838
9	4560	N/A	5673
10	4951	N/A	6084
11	5792	N/A	6719
12	5626	N/A	6823
13	4338	N/A	5446
14	3843	N/A	4824
15	4305	N/A	5045
16	4272	N/A	5329
17	4110	N/A	5347
18	4824	N/A	6705
19	5807	N/A	7026
20	5368	N/A	6988

b. –5°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	N/A	0
2	1000	N/A	1451
3	2593	N/A	4145
4	2391	N/A	4084
5	N/A	N/A	N/A
6	131	N/A	602
7	91	N/A	985
8	86	N/A	870
9	N/A	N/A	N/A
10	442	N/A	1035
11	336	N/A	1898
12	331	N/A	1280
13	N/A	N/A	N/A
14	189	N/A	703
15	75	N/A	1238
16	80	N/A	511
17	N/A	N/A	N/A
18	568	N/A	1437
19	165	N/A	1976
20	354	N/A	1393

c. –10°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	N/A	0
2	284	N/A	438
3	1211	N/A	2556
4	1649	N/A	3654
5	N/A	N/A	N/A
6	0	N/A	0
7	0	N/A	0
8	0	N/A	0
9	N/A	N/A	N/A
10	0	N/A	0
11	0	N/A	0
12	0	N/A	24
13	N/A	N/A	N/A
14	0	N/A	0
15	0	N/A	0
16	0	N/A	0
17	N/A	N/A	N/A
18	0	N/A	0
19	0	N/A	0
20	0	N/A	0

d. –20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	N/A	0
2	0	N/A	0
3	0	N/A	556
4	0	N/A	842
5	N/A	N/A	N/A
6	0	N/A	0
7	0	N/A	0
8	0	N/A	0
9	N/A	N/A	N/A
10	0	N/A	0
11	0	N/A	0
12	0	N/A	0
13	N/A	N/A	N/A
14	0	N/A	0
15	0	N/A	0
16	0	N/A	0
17	N/A	N/A	N/A
18	0	N/A	0
19	0	N/A	0
20	0	N/A	0

Table A6. Chemical composition of admixtures tested in Phase 1, Task C.

Mix no.	*Antifreeze admixture formulation*	*% Solids in sol.*	*Dosage (% CWT)*	*w/c Ratio*	*Slump (cm)*	*Remarks*
1	Control	0	0	0.48	4	Concrete temp at 18°C
2	KC1	30	2	0.48	4	Concrete temp at 18°C
3	KC1	30	6	0.48	7	Concrete temp at 18°C
4	KC1	30	8	0.48	9	Concrete temp at 18°C
5	Control	0	0	0.48	4	Concrete temp at 18°C
6	CCNDD-5	33	2	0.48	3	Concrete temp at 18°C
7	CCNDD-5	33	4	0.48	4	Concrete temp at 20°C
8	CCNDD-5	33	6	0.48	4	Concrete temp at 21°C
9	Control	0	0	0.48	3	Concrete temp at 18°C
10	CC-SONI/NMP	33	2	0.48	2	Concrete temp at 18°C
11	CC-SONI/NMP	33	4	0.48	3	Concrete temp at 21°C
12	CC-SONI/NMP	33	6	0.48	3	Concrete temp at 21°C
13	Control	0	0	0.48	3	Concrete temp at 18°C
14	CCD-NMP	33	2	0.48	2	Concrete temp at 18°C
15	CCD-NMP	33	4	0.48	2	Concrete temp at 18°C. Too-fast set.
16	CCD-NMP	33	6	0.48	4	Concrete temp at 18°C. Too-fast set.
17	Control	0	0	0.48	3	Concrete temp at 18°C
18	CC-NMP	33	2	0.48	3	Concrete temp at 18°C
19	CC-NMP	33	4	0.48	3	Concrete temp at 18°C
20	CC-NMP	33	6	0.48	1	Concrete temp at 23°C. Too-fast set.

Table A7. Compressive strength. Task 1D cured at various temperatures.

a. 20°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	4691	5293	5603
2	N/A	N/A	N/A
3	2855	5800	3489
4	3815	4178	4366
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	3230	5564	6437
8	5079	6163	6432
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	1893	3249	3131
4	2613	4494	4866
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	2490	4419	5116
8	1570	3725	4593
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A7 (cont'd). Compressive strength. Task 1D cured at various temperatures.

c. –10°C

Mix no.	Compressive strength (psi) Testing age (days) 7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	0	1325	1438
4	1307	2726	2895
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	355	1261	2165
8	0	1038	2320
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C

Mix no.	Compressive strength (psi) Testing age (days) 7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	664	247	98
4	162	126	667
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	0	39	123
8	0	0	691
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A8. Chemical composition of admixtures tested in Phase 1, Task D.

Mix no.	Antifreeze admixture formulation	% Solids in sol.	Dosage (% CWT)	w/c Ratio	Slump (cm)	Remarks
1	Control	0	0	0.48	5	
2	K_2CO_2 + lignosulfonate	30	6/0.5	0.43	7	10% water reduction.
3	K_2CO_2 + lignosulfonate	30	6/1.5	0.43	19	10% water reduction.
4	K_2CO_2 + lignosulfonate	30	6/1.5	0.38	1	20% water reduction.
5	Control	0	0	0.48	5	
6	$Ca(NO_2)_2 + NaNO_2$	30	1.5/1.5	0.48	5	
7	$Ca(NO_2)_2 + NaNO_2$	30	3/3	0.48	6	
8	$Ca(NO_2)_2 + NaNO_2$	30	5/5	0.48	4	
9	N/A	N/A	N/A	N/A	N/A	
10	N/A	N/A	N/A	N/A	N/A	
11	N/A	N/A	N/A	N/A	N/A	
12	N/A	N/A	N/A	N/A	N/A	
13	N/A	N/A	N/A	N/A	N/A	
14	N/A	N/A	N/A	N/A	N/A	
15	N/A	N/A	N/A	N/A	N/A	
16	N/A	N/A	N/A	N/A	N/A	
17	N/A	N/A	N/A	N/A	N/A	
18	N/A	N/A	N/A	N/A	N/A	
19	N/A	N/A	N/A	N/A	N/A	
20	N/A	N/A	N/A	N/A	N/A	

Table A9. Compressive strength. Task 1E cured at various temperatures.

a. 20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	4291	5546	5899
2	5220	5819	6248
3	4904	5461	5263
4	4649	5210	5843
5	4633	5222	5508
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	42	119	182
2	910	1783	2370
3	1227	1910	2428
4	927	1594	2211
5	1278	2137	2791
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

c. –10°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	0	0
2	0	31	178
3	40	91	309
4	43	84	271
5	70	152	348
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A10. Chemical composition of admixtures tested in Phase 1, Task E.

Mix no.	*Antifreeze admixture formulation*	*% Solids in sol.*	*Dosage (% CWT)*	*w/c Ratio*	*Slump (cm)*	*Remarks*
1	Control	0	0	0.48	3	
2	CM-42	30	4	0.48	4	
3	CM-42	30	6	0.48	3	
4	CM-48	30	4	0.48	3	
5	CM-48	30	6	0.48	3	
6	N/A	N/A	N/A	N/A	N/A	
7	N/A	N/A	N/A	N/A	N/A	
8	N/A	N/A	N/A	N/A	N/A	
9	N/A	N/A	N/A	N/A	N/A	
10	N/A	N/A	N/A	N/A	N/A	
11	N/A	N/A	N/A	N/A	N/A	
12	N/A	N/A	N/A	N/A	N/A	
13	N/A	N/A	N/A	N/A	N/A	
14	N/A	N/A	N/A	N/A	N/A	
15	N/A	N/A	N/A	N/A	N/A	
16	N/A	N/A	N/A	N/A	N/A	
17	N/A	N/A	N/A	N/A	N/A	
18	N/A	N/A	N/A	N/A	N/A	
19	N/A	N/A	N/A	N/A	N/A	
20	N/A	N/A	N/A	N/A	N/A	

Table A11. Compressive strength. Task 1F cured at various temperatures.

a. 20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	5899	6149	N/A
2	5630	6055	N/A
3	6078	6375	N/A
4	6319	6427	N/A
5	5541	6218	N/A
6	3551	3810	N/A
7	6602	7267	N/A
8	N/A	N/A	N/A
9	6814	7545	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	152	49	N/A
2	870	1352	N/A
3	655	956	N/A
4	648	1266	N/A
5	797	1120	N/A
6	94	0	N/A
7	882	1190	N/A
8	1546	2098	N/A
9	839	1219	N/A
10	1159	1502	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A11 (cont'd).

c. –10°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	45	0	N/A
2	146	52	N/A
3	168	74	N/A
4	118	0	N/A
5	163	41	N/A
6	0	0	N/A
7	281	278	N/A
8	N/A	N/A	N/A
9	120	353	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A12. Chemical composition of admixtures tested in Phase 1, Task F.

Mix no.	Antifreeze admixture formulation	% Solids in sol.	Dosage (% CWT)	w/c Ratio	Slump (cm)	Remarks
1	Control (with WRDA-19)	0	0.56	0.40	0	Contains 12 fl oz of HRWR/CWT
2	PolarSet (with WRDA-19)	4	0.61	0.40	0	Contains 13 fl oz of HRWR/CWT
3	PolarSet (with WRDA-19)	6	0.70	0.40	0	Contains 15 fl oz of HRWR/CWT
4	PolarSet (with AA1-D)	4	0.56	0.40	0	Contains 13 fl oz of HRWR/CWT
5	PolarSet (with AA1-D)	6	0.56	0.40	0.5	Contains 13 fl oz of HRWR/CWT
6	Control (with WRDA-19)	0	0.70	0.40	0	Contains 15 fl oz of HRWR/CWT
7	DCI (with WRDA-19)	4	0.70	0.40	0	Contains 15 fl oz of HRWR/CWT. Too-fast set.
8	DCI (with WRDA-19)	6	0.70	0.40	0	Contains 15 fl oz of HRWR/CWT
9	DCI (with AA1-D)	4	0.56	0.40	0	Contains 13 fl oz of HRWR/CWT. Too-fast set.
10	DCI (with AA1-D)	6	0.56	0.40	0	
11	N/A	N/A	N/A	N/A	N/A	
12	N/A	N/A	N/A	N/A	N/A	
13	N/A	N/A	N/A	N/A	N/A	
14	N/A	N/A	N/A	N/A	N/A	
15	N/A	N/A	N/A	N/A	N/A	
16	N/A	N/A	N/A	N/A	N/A	
17	N/A	N/A	N/A	N/A	N/A	
18	N/A	N/A	N/A	N/A	N/A	
19	N/A	N/A	N/A	N/A	N/A	
20	N/A	N/A	N/A	N/A	N/A	

Table A13. Compressive strength. Task 1Ga cured at various temperatures.

a. 20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	1214	1722	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	0	N/A
2	1467	2413	N/A
3	875	1590	N/A
4	677	1482	N/A
5	1483	1910	N/A
6	1377	1950	N/A
7	874	1108	N/A
8	950	942	N/A
9	1628	2494	N/A
10	818	1349	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

c. –10°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A14. Chemical composition of admixtures tested in Phase 1, Task Ga.

Mix no.	*Antifreeze admixture formulation*	*Dosage (% CWT)*	*w/c Ratio*	*Remarks*
1	Control	0	0.45	Mix too fluid.
2	$Ca(NO_2)_2$ /propylene glycol	3/3	0.40	Moderately fast set.
3	$Ca(NO_2)_2$ /propylene glycol	4.2/1.8	0.40	Faster set.
4	$Ca(NO_2)_2$ /propylene glycol	1.8/4.2	0.40	Moderately fast set.
5	Urea/$Ca(NO_2)_2$	1.5/4.5	0.40	
6	Urea/$Ca(NO_2)_2$	4.5/1.5	0.40	Easely workable.
7	$Ca(NO_2)_2$ /Daratard	6/0.26	0.42	Too-rapid set.
8	$Ca(NO_2)_2$ /Daratard + microsilica (25% PC substitution)	6/0.78	0.42	Gritty mix; clay-like: moldable but not bleeding.
9	$Ca(NO_2)_2$ /cane sugar	6/0.1	0.40	Soft and workable; not bleeding. Moderate set at 40 minutes.
10	$Ca(NO_2)_2$ /latex	6/7.5	0.45	Sticky; too-fast set.
11	N/A	N/A	N/A	
12	N/A	N/A	N/A	
13	N/A	N/A	N/A	
14	N/A	N/A	N/A	
15	N/A	N/A	N/A	
16	N/A	N/A	N/A	
17	N/A	N/A	N/A	
18	N/A	N/A	N/A	
19	N/A	N/A	N/A	
20	N/A	N/A	N/A	

Table A15. Compressive strength. Task 1Gb cured at various temperatures.

a. 20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	6027	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	953	N/A	N/A
2	1936	N/A	N/A
3	1000	N/A	N/A
4	1456	N/A	N/A
5	851	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A15 (cont'd). Compressive strength. Task 1Gb cured at various temperatures.

c. –10°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A16. Chemical composition of admixtures tested in Phase 1, Task Gb.

Mix no.	*Antifreeze admixture formulation*	*Dosage (% CWT)*	*w/c Ratio*	*Remarks*
1	Control	0	0.43	
2	DCI	4	0.43	
3	DCI/propylene glycol	3.6/0.4	0.43	
4	DCI/propylene glycol	3.2/0.8	0.43	
5	DCI/propylene glycol	2.8/1.2	0.43	
6	N/A	N/A	N/A	
7	N/A	N/A	N/A	
8	N/A	N/A	N/A	
9	N/A	N/A	N/A	
10	N/A	N/A	N/A	
11	N/A	N/A	N/A	
12	N/A	N/A	N/A	
13	N/A	N/A	N/A	
14	N/A	N/A	N/A	
15	N/A	N/A	N/A	
16	N/A	N/A	N/A	
17	N/A	N/A	N/A	
18	N/A	N/A	N/A	
19	N/A	N/A	N/A	
20	N/A	N/A	N/A	

Table A17. Compressive strength. Task 1Gc cured at various temperatures.

a. 20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	1714	1744	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	0	N/A
2	1217	1862	N/A
3	1495	2392	N/A
4	1267	2135	N/A
5	1757	2357	N/A
6	N/A	N/A	N/A
7	659	1189	N/A
8	1261	2543	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

c. –10°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	0	N/A
2	0	0	N/A
3	84	701	N/A
4	520	1017	N/A
5	133	332	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	569	1337	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A18. Chemical composition of admixtures tested in Phase 1, Task Gc.

Mix no.	*Antifreeze admixture formulation*	*Dosage (% CWT)*	*w/c Ratio*	*Remarks*
1	Control	0	0.42	Normal plasticity.
2	$Ca(NO_2)_2$ /propylene glycol	3/3	0.39	Normal plasticity.
3	Urea/$Ca(NO_2)_2$	1.5/4.5	0.42	Less plastic than previous mix.
4	Urea/$Ca(NO_2)_2$	4.5/1.5	0.42	Very soft mix; little bleeding.
5	$Ca(NO_2)_2$ /cane sugar	6/0.4	0.42	Rapid set.
6	$Ca(NO_2)_2$ /cane sugar	9/0.4	0.42	Excessive rapid set.
7	$Ca(NO_2)_2$ /cane sugar + microsilica (25% cement substitution)	6/0.3	0.45	Too-rapid set.
8	K_2CO_2 /cane sugar	6/0.4	0.42	Normal plasticity kept by remixing at intervals.
9	N/A	N/A	N/A	
10	N/A	N/A	N/A	
11	N/A	N/A	N/A	
12	N/A	N/A	N/A	
13	N/A	N/A	N/A	
14	N/A	N/A	N/A	
15	N/A	N/A	N/A	
16	N/A	N/A	N/A	
17	N/A	N/A	N/A	
18	N/A	N/A	N/A	
19	N/A	N/A	N/A	
20	N/A	N/A	N/A	

Table A19. Compressive strength. Task 1H cured at various temperatures.

a. 20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	5550	6088	6681
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

b. –5°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	231	254	478
2	2947	4249	5022
3	1912	3150	4186
4	2516	3711	4702
5	1723	3046	4238
6	2964	4183	5043
7	2098	3442	4385
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A19 (cont'd).

c. –10°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	0	45	110
2	60	93	432
3	112	42	1032
4	41	384	126
5	64	275	766
6	55	87	503
7	162	373	754
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

d. –20°C.

	Compressive strength (psi)		
	Testing age (days)		
Mix no.	7	14	28
1	N/A	N/A	N/A
2	N/A	N/A	N/A
3	N/A	N/A	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	N/A	N/A	N/A
8	N/A	N/A	N/A
9	N/A	N/A	N/A
10	N/A	N/A	N/A
11	N/A	N/A	N/A
12	N/A	N/A	N/A
13	N/A	N/A	N/A
14	N/A	N/A	N/A
15	N/A	N/A	N/A
16	N/A	N/A	N/A
17	N/A	N/A	N/A
18	N/A	N/A	N/A
19	N/A	N/A	N/A
20	N/A	N/A	N/A

Table A20. Chemical composition of admixtures tested in Phase 1, Task H.

Mix no.	*Antifreeze admixture formulation*	*Dosage (% CWT)*	*w/c Ratio*	*Slump (cm)*	*Remarks*
1	Control	0	0.43	6	
2	DP	6	0.43	6	
3	DP	8	0.43	6	
4	DPT	6	0.43	6	
5	DPT	8	0.43	5	
6	DPTC	6	0.43	5	
7	DPTC	8	0.43	5	
8	N/A	N/A	N/A	N/A	
9	N/A	N/A	N/A	N/A	
10	N/A	N/A	N/A	N/A	
11	N/A	N/A	N/A	N/A	
12	N/A	N/A	N/A	N/A	
13	N/A	N/A	N/A	N/A	
14	N/A	N/A	N/A	N/A	
15	N/A	N/A	N/A	N/A	
16	N/A	N/A	N/A	N/A	
17	N/A	N/A	N/A	N/A	
18	N/A	N/A	N/A	N/A	
19	N/A	N/A	N/A	N/A	
20	N/A	N/A	N/A	N/A	

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APPENDIX B: THERMAL HISTORY OF SLAB-WALL PROTOTYPE

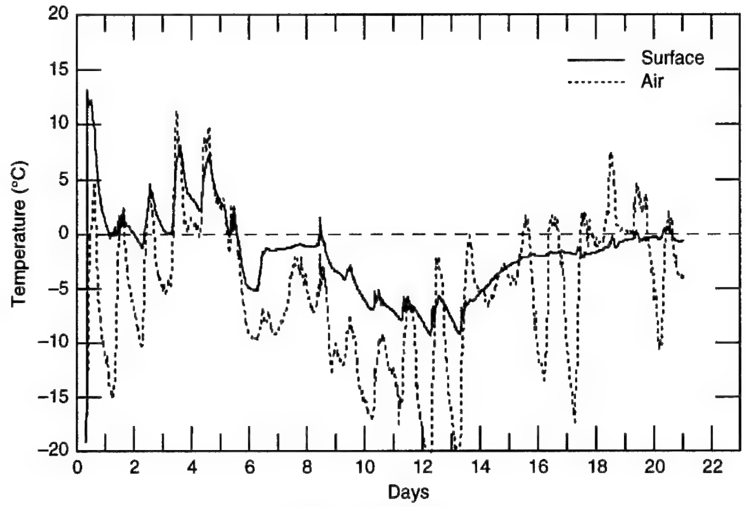


Figure B1. Slab.

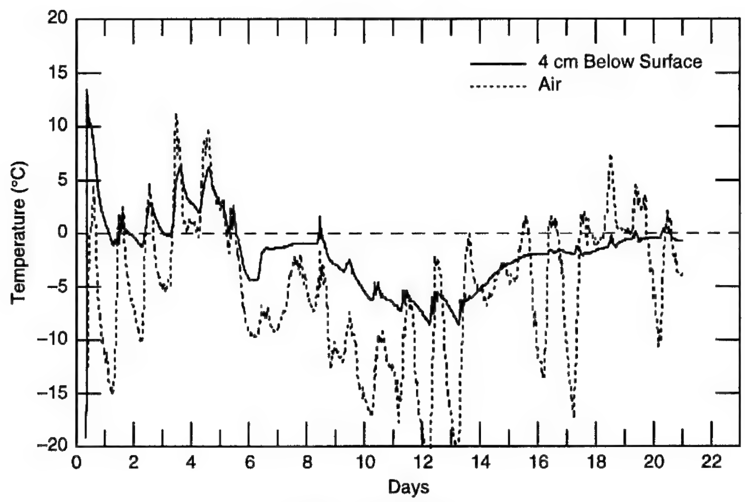


Figure B2. Slab.

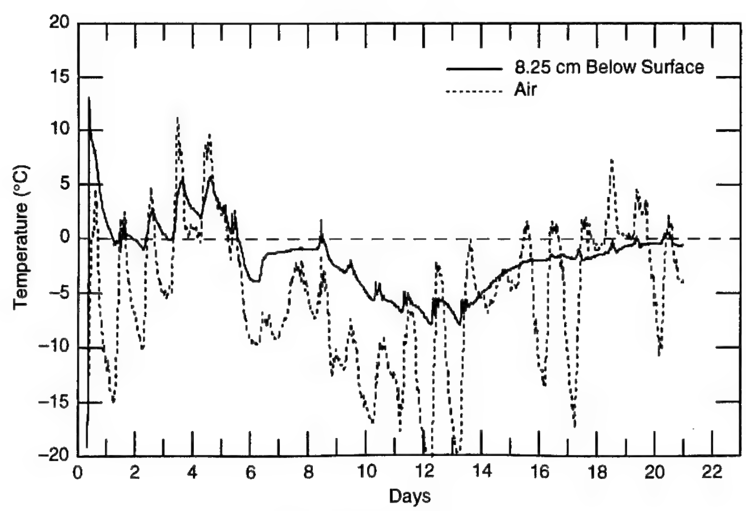
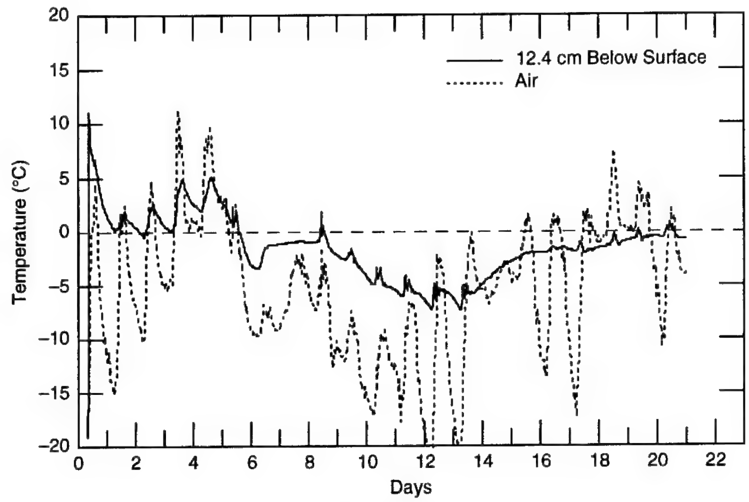


Figure B3. Slab.



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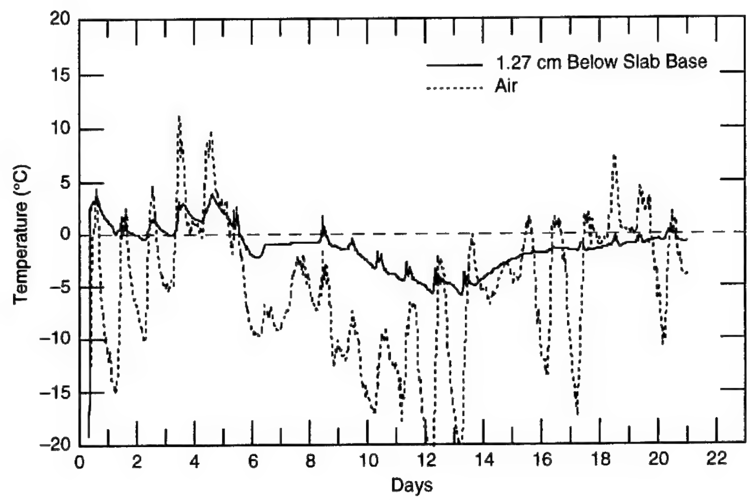


Figure B5. Slab.

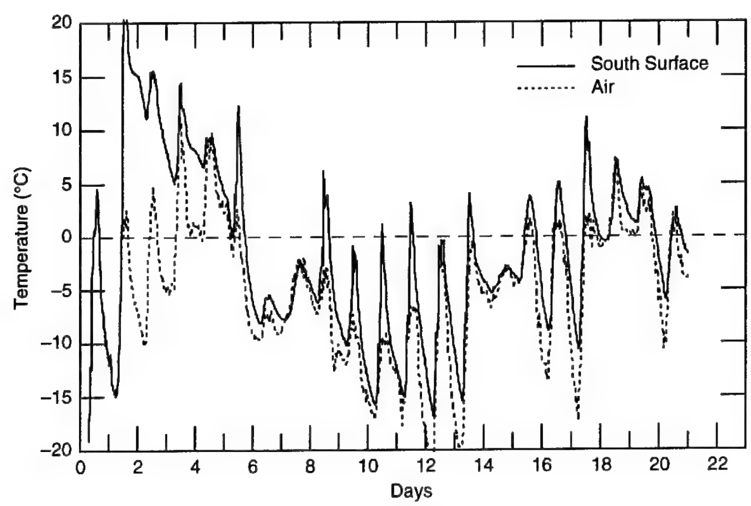
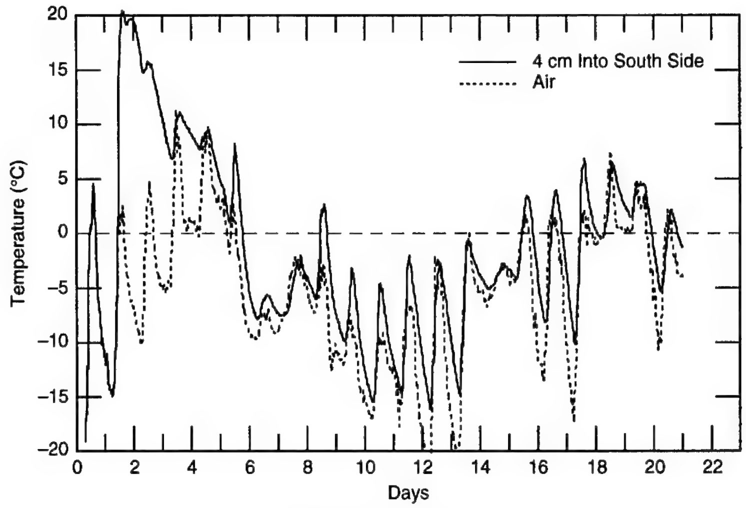


Figure B6. Wall.



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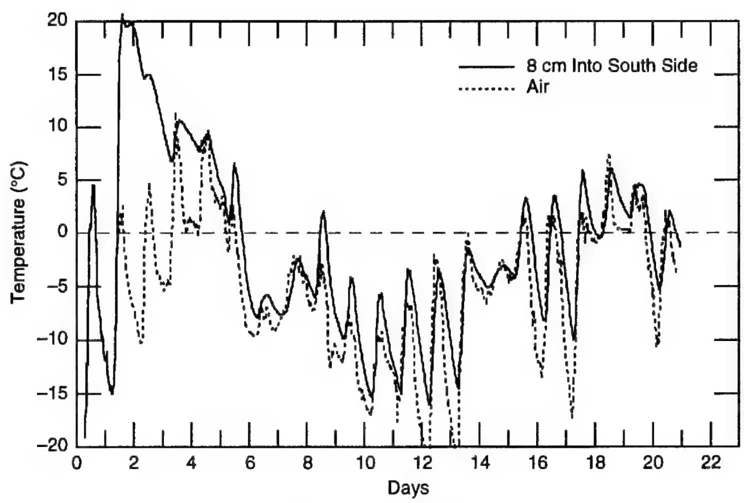

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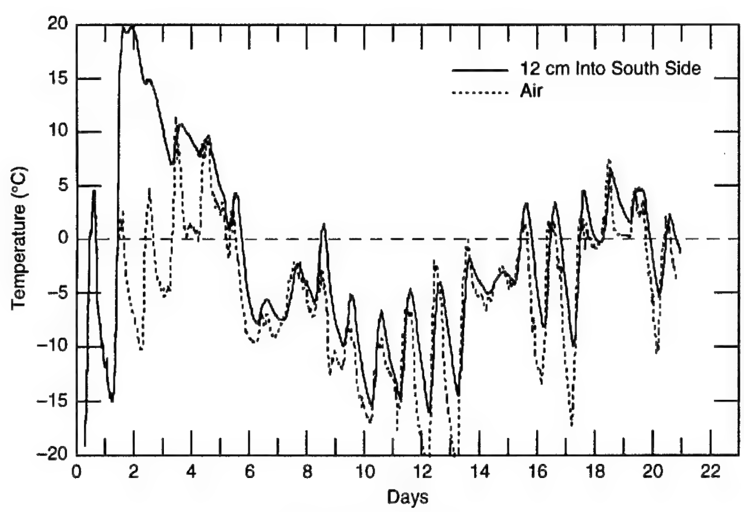

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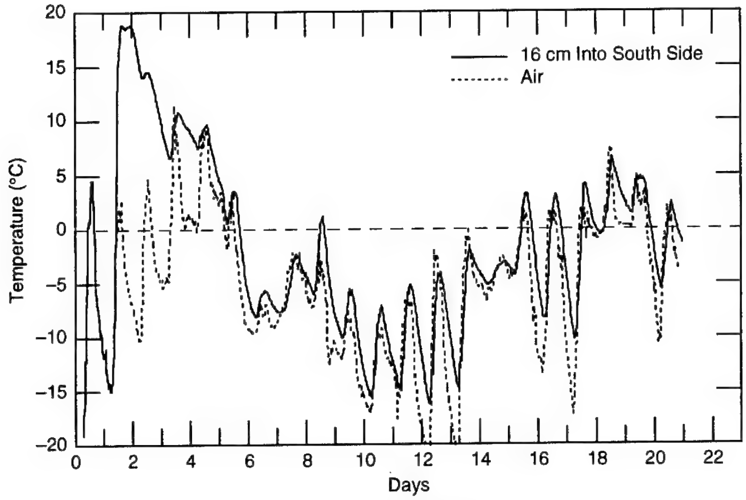

Figure B10. Wall.

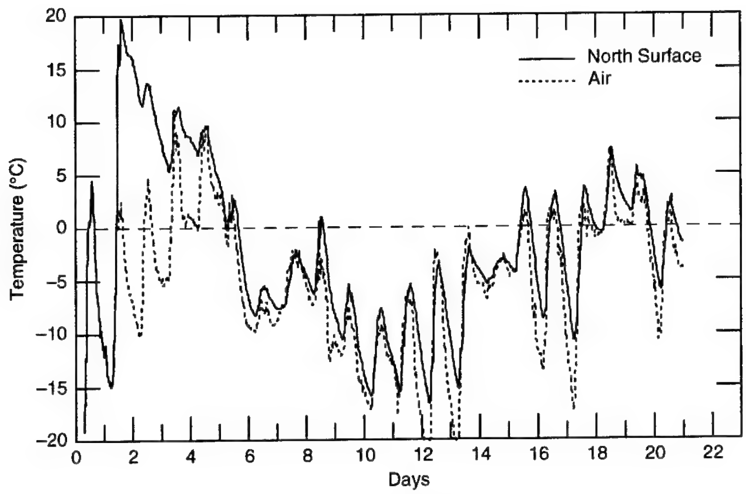

Figure B11. Wall.

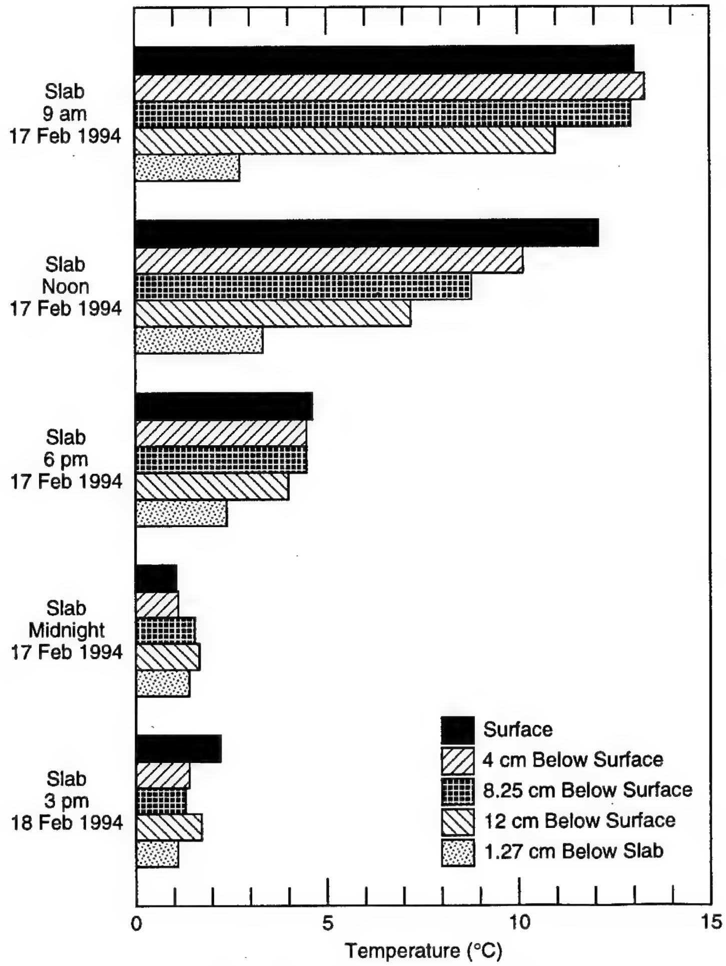

Figure B12. Thermal record, 17–18 February.

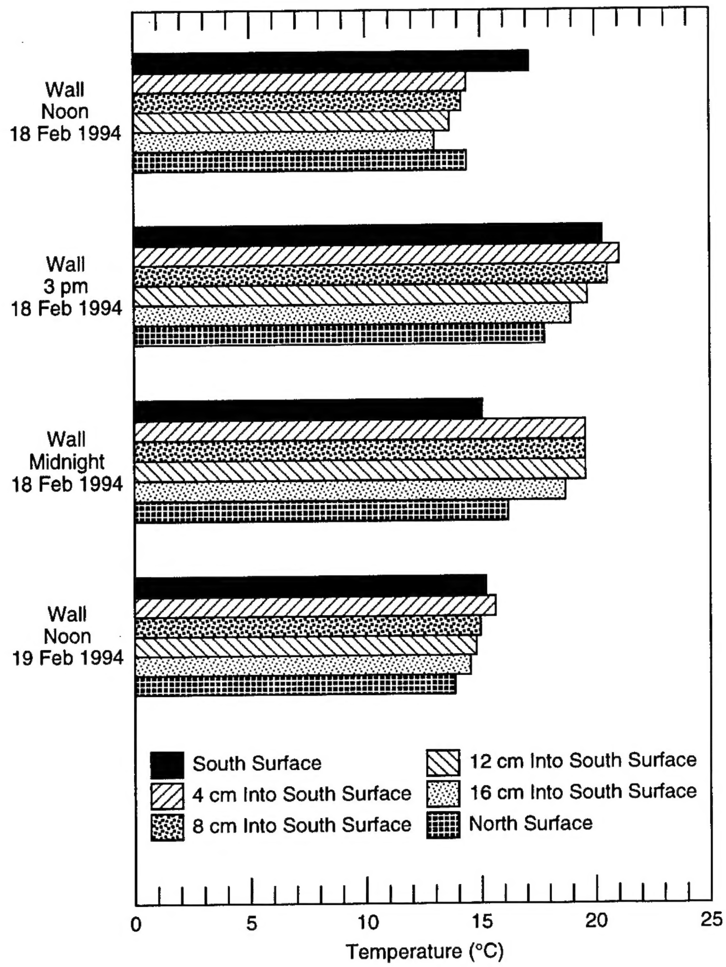

Figure B13. Thermal record, 18–19 February.

APPENDIX C: PROPOSED CHANGES TO USACE GUIDANCE

Example document to be changed: CEGS 03300 (September 1995)
Section: 2.3 Chemical Admixtures

Change #1

Insert "Antifreeze Admixtures" as number 2.3.7 using the text below, and renumber the section on "Other Chemical Admixtures" to be number 2.3.8.

Suggested Insertion

Antifreeze admixtures

An antifreeze admixture shall be able to promote the strength of concrete cured at –5°C ±1°C to reach a minimum seven-day strength of 40% of the strength of an equivalent admixture-free concrete cured at 20°C at the same age, and a minimum 14-day strength of 60% of the strength of an equivalent admixture-free concrete cured at 20°C at the same age. The test specimens shall be standard cylinders 7.6 cm in diameter or larger. The concrete shall be mixed and cylinders cast at room temperature (about 20°C). The specimens to be cured at –5°C shall be brought into the cold chamber no more than 40 minutes after water has been added to the cement during mixing. The cold chamber shall be able to cool the center of mass of the cylinders to 0°C or lower within three hours, and to –4°C within eight hours. The temperature of the cylinders shall be measured with embedded thermocouples on replicate specimens. On the day of the compressive strength test, the cylinders shall be moved from the cold room to a warm room at about 20°C. The temperature at the cylinders' center of mass shall be allowed to reach 5°C before being compression tested. The cylinders shall be compression tested within one hour of reaching 5°C.

Except for the strength requirements, the admixture shall meet the physical requirements set forth in ASTM C 494 for an admixture Type C.

For cast-in-place concrete, the admixture shall not contain more than 2% of calcium chloride by weight of cement. For prestressed concrete, the admixture shall not contain any chlorides, except in trace amounts.

The admixture may add alkalis only to the extent that the total mixture alkali content does not exceed 0.6% by weight of cement.

Change #2

Rewrite section 3.8.3, "Cold Weather Requirements," as follows:

There are two possible protection measures that can be used when the ambient temperature drops below 0°C. The first measure entails thermal protection and the second method entails chemical protection. Whichever method is chosen, it must be approved by the Contracting Officer.

<u>*Thermal Protection*</u> may be chosen if freezing temperatures are anticipated before the expiration of the specified curing period. The ambient temperature of the air where concrete is to be placed and the temperature of surfaces to receive concrete shall not be less than 5°C (40°F). Heating of the mixing water or aggre-

gates will be required to regulate the concrete placing temperature. Materials entering the mixer shall be free from ice, snow, or frozen lumps. Upon written approval, an accelerating admixture conforming to ASTM C 494, Type C or E, may be used, provided it contains no calcium chloride.

Antifreeze admixtures may be chosen in lieu of thermal protection provided the concrete temperature, at it coldest section, does not dip below the lowest protection capability of the admixture. The admixture shall conform to the requirements of section 2.3.7 of this document.

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11. SUPPLEMENTARY NOTES

For conversion of SI units to non-SI units of measurement consult ASTM Standard E380-93, *Standard Practice for Use of the International System of Units,* published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

Available from NTIS, Springfield, Virginia 22161

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)

The goal of this project was to develop a chemical admixture that would reduce the need for wintertime thermal protection of freshly placed concrete. Chemicals were investigated for their ability to promote strength gain in concrete cured below 0°C. The project was carried out in five phases. Phase 1 evaluated existing and new admixtures. Phase 2 measured the effect of promising chemicals on concrete properties. Phases 3 and 4 tested the practicality of using the new technology/admixture in the field. Phase 5 disseminated the findings through an Army conference and through the development of this report, in addition to normal W.R. Grace advertising channels. Laboratory strength tests established that two prototype admixtures were capable of protecting concrete down to –5°C. Results from other laboratory tests show that the chemicals pose no harm to the concrete or embedded ferrous metals. Concrete containing the prototype admixtures passes standard freeze–thaw tests, does not shrink unusually, does not contain harmful alkalis, and does not produce irregular hydration products. Field tests clearly demonstrated that working with these new admixtures requires no new skills. The concrete can be mixed at lower temperatures, saving energy. The admixtures are easily dosed into the mixing trucks, as is normal practice today, and concrete is finished in the usual manner. Estimates show that the two prototype admixtures can extend the construction season by as much as three months in the contiguous United States. The prototype has proved that low-temperature admixtures are possible. The industry partner sees the need to develop admixtures that will work to –10°C before going commercial with this technology.

14. SUBJECT TERMS

Antifreeze admixtures, Cold concrete, Cold regions construction, Cold weather concrete, Concrete admixtures, Freeze-point depression, Low-temperature strength, Thermal protection, Winter construction

15. NUMBER OF PAGES

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